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COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM. (U)  
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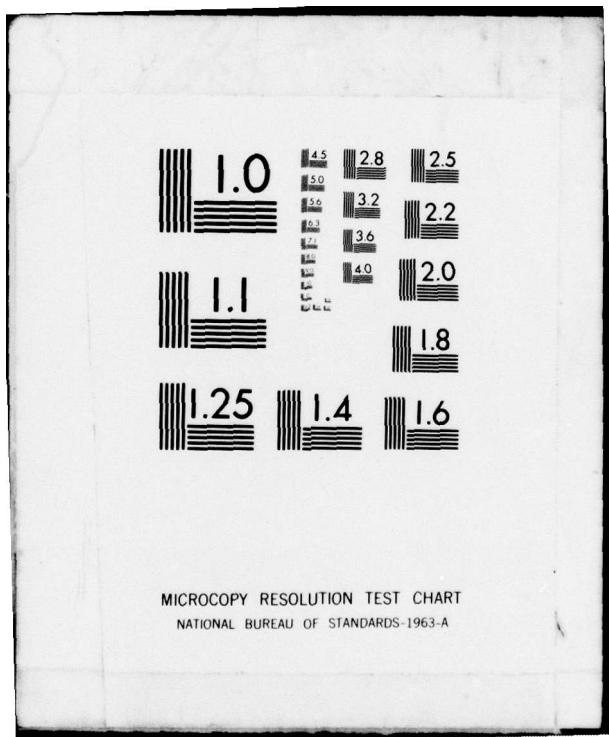
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NRL Memorandum Report 4080	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) ⑥ COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM, ANNUAL PROGRESS REPORT FOR THE PERIOD 1 SEPTEMBER 1977 - 31 AUGUST 1978 A041740		5. TYPE OF REPORT & PERIOD COVERED Progress Report 1 September 1977 31 August 1978
7. AUTHOR(s) ⑩ F. A. Smidt, Jr. L. A. Beach Division		6. PERFORMING ORG. REPORT NUMBER ⑪ 4 Oct 79
9. PERFORMING ORGANIZATION NAME AND ADDRESS Naval Research Laboratory Washington, DC 20375 ⑫ 72		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NRL Problems M01-22, M01-27, H01-83, H01-70 and H01-67
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research Arlington, Virginia 22217		12. REPORT DATE October 4, 1979
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) ⑭ NRL-MR-4080		13. NUMBER OF PAGES 71
15. SECURITY CLASS. (of this report) UNCLASSIFIED		
15a. DECLASSIFICATION/DOWNGRADING SCHEDULE		
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) ⑨ Annual progress rept. 1 Sep 77-31 Aug 78,		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Computer modeling Energy deposition Deuterium diffusion Heavy ion irradiation Dislocations Heavy ion radiation damage Dislocation loops He, D-elastic scattering measurements Electron microscopy Helium diffusion (Continues)		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The Cooperative Radiation Effects Simulation Program (CORES) is a collaborative effort of the Material Science and Technology and Radiation Technology Divisions of NRL Material and Radiation Science and Technology Area. The goal of the research is to provide the theoretical and experimental bases for understanding the mechanisms of nuclear radiation damage of metals, as well as a theoretical insight into energy deposition processes. In this the Van de Graaff and Cyclotron are used to simulate rapidly the radiation damage produced over long periods in reactor neutron environments. (Continues)		

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S/N 0102-014-6601

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## 19. Key Words (Continued)

Hydrogen diffusion  
 Interstitials in metals  
 Light ion irradiations  
 Metal radiation damage  
 Molybdenum  
 Neutron radiation damage  
 Nickel alloys  
 Nickel ion beams  
 Niobium  
 Nb-1%Zr  
 Primary knockons  
 Radiation damage  
 Radiation damage simulation  
 X-ray diffraction measurements of dislocation density

Radiation-induced defects  
 Radiation-induced displacements  
 Refractory metal alloys  
 Simulation of neutron irradiations  
 Swelling  
 Temperature dependence of void formation  
 Transport theory  
 TZM  
 Vacancies in metals  
 Vanadium  
 Van de Graaff bombardments  
 Voids  
 Void growth

## 20. Abstract (Continued)

Progress for the period, 1 September 1977 to 31 August 1978, includes: (1) development of a method for calculating energy deposition profiles from range distributions, (2) development of a method to estimate energy deposition in ion bombardments which incorporate transport of energy downstream by primary knockon atoms, (3) an investigation of the influence of 1 atomic percent Al, Mo, Si and Ti, respectively, to the swelling behavior of binary nickel alloys under neutron irradiation; (4) an investigation of swelling in refractory metals and alloys (Mo, Nb, V, TZM, Nb-1% Zr and V-20% Ti) at 625°C for fast neutron fluences to  $5.4 \times 10^{22} \text{ n/cm}^2$ ; (5) an investigation of deuterium trapping in radiation damaged aluminum and stainless steel using elastic scattering techniques to measure the increase in dislocation density produced by radiation damage in copper single crystals.

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## COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report  
for the period  
1 September 1977 - 31 August 1978

### Program Description

The Cooperative Radiation Effects Simulation Program (CORES) was initiated voluntarily by five Branches from the Engineering Materials (now the Material Science and Technology Division) and the Radiation Technology Division of NRL on the basis of their common interests in the problems of simulating radiation damage in metals. The program promotes the exchange of information, discussion of problems, and the pursuit of collaborative research efforts. Annually a written report is prepared containing those portions of the work of the participating Branches which are judged to be of interest to the damage simulation problem. The major portion of the work is sponsored by the Office of Naval Research. Since research findings which apply to the objectives of one sponsor may also be of interest to others, the overall progress related to damage simulation is included in the written report. Several of the participating Branches have independent programs on other aspects of the radiation damage problem; when results obtained in these programs are judged to be of interest to CORES participants they may also be included, informally, in the CORES program review.

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## COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report  
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1 September 1977 - 21 August 1978

### SUMMARY

#### I. RADIATION DAMAGE THEORY

##### A. Use of Range Distribution to Approximate Energy Distributions

Range distributions can be used to estimate the distribution in energy which develops in an ion beam as it penetrates a target. Several variants of this approach are discussed and applied to the calculation of energy deposition profiles.

##### B. Approximation for Energy Deposition in Ion Beam Bombardment

A method is developed for estimating energy deposition in ion bombardment. By making extensive use of a table of range versus energy, it is possible to estimate the energy transport effects due to primary knock-on atoms depositing energy downstream from the sites of their creation.

#### II. HEAVY ION AND NEUTRON DAMAGE STUDIES

##### A. The Effect of Alloy Additions on the Swelling of Neutron Irradiated Nickel

The void and dislocation structures in pure nickel and the binary alloys Ni-1 at.% Al, Ni-1 at.% Mo, Ni-1 at.% Si, and Ni-1 at.% Ti were examined following neutron irradiation to  $2.2 \times 10^{22}$  n/cm<sup>2</sup> ( $E > 0.1$  MeV) at 455°C. The dislocation structures of all the materials were similar, consisting of tangles of line dislocations, many of which intersected voids. Comparing the void formation of the alloys to that in the pure nickel, the following observations were made: the Al addition did not significantly affect the void formation, except to make it more homogeneous and to narrow the size distribution; the Mo and Ti both increased the swelling by about a factor of two, with the Mo increasing the void number density and the Ti increasing the void size; the Si decreased the swelling by about a factor of ten, by reducing the void size. These results differ significantly from those of an earlier ion irradiation study of the same alloys, in which all of these alloying elements reduced or eliminated the void formation. The reason for these differences is most likely related to different void nucleation mechanisms being important in the two types of irradiation.

## B. The Microstructure of Neutron Irradiated Refractory Metals and Alloys

The refractory metals Mo, Nb, and V and the alloys TZM, Nb-1%Zr and V-20%Ti were examined by transmission electron microscopy following irradiation at 650°C to fluences of  $5.4 \times 10^{22}$  n/cm<sup>2</sup>, E>0.1 MeV (all materials except V-20%Ti) and  $8.4 \times 10^{22}$  n/cm<sup>2</sup>, E>0.1 MeV (Mo and V-20% Ti). Voids representing significant volume swelling were observed in the Mo (2.3 - 3.0%), TZM (4.0%), Nb (1.5%) and V (3.6%). Some precipitation occurred during irradiation in all materials except the Mo and V. The results indicate that void swelling can probably be controlled at moderate levels at this temperature in refractory alloys. Degradation of mechanical properties due to precipitation of interstitial impurities may, however, be a more serious problem.

## III. LIGHT ION BEAM STUDIES

### A. Deuterium Retention in Aluminum and Stainless Steel With and Without Helium Loading

Thin samples of aluminum (1.2 mils) and stainless steel (0.6 mils) were implanted with deuterium both with and without prior implantations of helium. These samples were then profiled to determine concentrations of helium and deuterium by elastic scattering techniques. Analysis of these measurements show deuterium was retained in aluminum foils both with and without previous helium implantations but deuterium was retained in stainless steel only with prior helium loading indicating enhanced deuterium retention with predamage in stainless steel.

### B. X-ray Diffraction Measurements of Low-Level Radiation Damage

X-ray diffraction gives a quantitative measure of early stages of radiation damage in metals. Alpha-particle irradiations of  $2 \times 10^{22}$ ,  $4 \times 10^{22}$ , and  $6 \times 10^{22}$  C/cm<sup>2</sup> in copper single crystals show an apparent threshold of little damage up to  $2 \times 10^{22}$  C/cm<sup>2</sup> and then a nearly linear increase of dislocations up to  $6 \times 10^{22}$  C/cm<sup>2</sup>. Preliminary experiments with polycrystalline copper, not discussed in this report, seem to show similar results.

## COOPERATIVE RADIATION EFFECTS SIMULATION PROGRAM

Annual Progress Report  
for the Period  
1 September 1977 - 21 August 1978

### INTRODUCTION

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This report summarizes annual research accomplishments of the inter-divisional cooperative program now in its eighth year. The goal of this program has been to use ion simulation techniques to increase the understanding of neutron damage of materials for advanced nuclear systems. Light and heavy ion bombardment techniques are used to represent neutron damage in order to accelerate research and to permit an evaluation of critical parameters which might not be feasible using nuclear reactors as test facilities. In this program the techniques applied have advanced along with our growing understanding of radiation damage of materials. To complement the ion damage simulation are other programs such as theoretical evaluations of atomic collisions, computation of energy deposition, and parallel experiments using nuclear reactors. The productivity of the CORES Program is illustrated by a full list of technical papers and presentations in the last section.

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Note: Manuscript submitted July 12, 1979

## RESEARCH PROGRESS

### I. RADIATION DAMAGE ANALYSIS

#### A. Use of Range Distributions to Approximate Energy Distributions

(Irwin Manning, Radiation-Matter Interaction Branch,  
Radiation Technology Division)

##### Introduction

Consider a uniform ion-beam of energy  $E_B$  bombarding an amorphous-target in slab geometry, with the beam parallel to the x-axis and the target surface located at  $x = 0$ . We are often interested in the single-particle flux density  $\phi(E, x)$  which gives the distribution in energy which develops in the ion beam when it penetrates to a distance  $x$ :

$$\phi(E, x) dE = \text{number of beam atoms with energy } dE \text{ about } E \text{ which cross, per unit bombarding flux per unit time, a perpendicular unit area at the depth } x. \quad (1)$$

According to this definition,  $\phi$  is normalized to unit bombarding flux:

$$\int_0^{\infty} \phi(E, 0) dE = 1. \quad (2)$$

Estimates of this flux density are difficult to obtain, but estimates are much more readily available for the range distribution  $f(x, E_B)$ ,

$$f(x) dx = \text{number of beam atoms per unit perpendicular area per unit bombarding flux per unit time coming to rest at the depth } dx \text{ about } x. \quad (3)$$

For example, in addition to this range distribution, reference 1 provides for a large variety of beam-target combinations, tables, giving as a function of beam energy, the total range  $R(E_B)$ , the projected range

$$R_p(E_B) = \langle x \rangle = \int_0^{\infty} f(x) dx, \quad (4)$$

and the range straggling  $\alpha(E_B)$ , where

$$\alpha^2(E_B) = \langle (x - x)^2 \rangle = \int_0^\infty (x - R_p)^2 f(x) dx. \quad (5)$$

According to the above definition, the range distribution is normalized to unity:

$$\int_0^\infty f(x) dx = 1. \quad (6)$$

This paper concerns a class of methods for using the range distribution  $f(x)$  to approximate the energy distribution  $\phi(E, x)$ . Usually one will utilize the estimate as the integrand inside an integral, which will tend to smooth out errors in  $\phi(E, x)$ . Section VI below discusses the employment of this estimate to obtain energy deposition profiles.

### Energy Distribution from the Range Distribution

We ignore the angular spread which develop in the penetrating beam as it comes to a stop, assuming that the particle velocities are always parallel to the  $x$ -axis. Consider the flux of particles  $f(x_2, dx_2)$  which will stop at  $dx_2$  about  $x_2$ . We make a model in which there are no range fluctuations (in the ordinary sense) among beam particles. In reality, different beam particles undergo different fluctuations in range, but on the average the beam obeys the range-stopping function  $R_p(E)$ . These fluctuations are removed in our model, and instead, fluctuations are introduced into the model by assigning different range-stopping functions,  $\tilde{R}_p(E, x_2)$  to different beam atoms, picked in such a way that the model has the range distribution  $f(x_2)$ . For the above flux of atoms which will stop in  $dx_2$  about  $x_2$ , consider their energy at some  $x_1 < x_2$ . The fluctuations in our model are such that all of these atoms, when at  $x = x_1$ , lie within a small energy interval  $dE$ , about the energy  $E_1$ . There is a one-to-one correspondence between those beam atoms which have energy  $E_1$  at  $x$  equal to  $x_1$  and those atoms which will stop at  $x$  equal to  $x_2$ . We have

$$\phi(E_1, x_1) dE_1 = f(x_2) dx_2, \quad (7)$$

where  $E_1$  is determined by  $x_1$  and  $x_2$  (and, implicitly,  $E_B$ ):

$$x_2 = x_1 + R_p(E_1, x_2) \quad (8)$$

which gives  $E_1$  as a function of  $x_1$  and  $x_2$ :

$$E_1 = E_1(x_1, x_2). \quad (9)$$

Once a specific model giving the altered range-stopping function  $R_p(E, x_2)$  is chosen,  $E_1(x_1, x_2)$  is determined by equation (8) and equation (7) then gives an estimate of the energy distribution. For a well-behaved function  $E_1(x_1, x_2)$ , this estimate is exact in the limit  $x_1 \rightarrow x_2$ , so we expect this estimate to be a good one for  $x_2 - x_1$  "small".

One easily verifies that the above estimate (7) obeys an identity satisfied by the true energy distribution. Consider the particle current

$$\vec{j}(\vec{r}) = \int_0^\infty \vec{\phi}(E, \vec{r}) dE, \quad (10)$$

where  $\vec{\phi}$  is the reactor flux corresponding to scalar flux (1). Gauss' law expresses the conservation of beam particles as

$$\operatorname{div} \vec{j} = -\rho(r), \quad (11)$$

where  $\rho$  is the volume density of stopping particles. For our slab geometry, equations (10) and (11) become

$$\partial/\partial x \ j(x) = -f(x) \quad (12)$$

where

$$j(x_1) = \int_{\text{all allowed } E_1} dE_1 \phi(x_1, x) \quad (13)$$

The estimate (4) gives

$$j(x_1) = \int_0^\infty dx_2 f(x_2), \quad (14)$$

which obeys the identity (12).

We now go on to consider specific models for  $\tilde{R}_p(E, x_2)$  and, correspondingly,  $E_1(x_1, x_2)$ .

#### Case A: No Fluctuations for $x > x_1$

Here we assume that different beam particles have different range-stopping functions  $\tilde{R}_p(E_1, x_2)$  only for  $x < x_1$ . For  $x > x_1$  we have, for all  $x_2$ ,

$$\begin{aligned} \tilde{R}_p(E_1, x_2) &= R_p(E_1), \\ x &> x_1. \end{aligned} \quad \left. \right\} \quad (15)$$

Equation (8) becomes

$$x_2 = x_1 + R_p(E_1) \quad (16)$$

As will be discussed in Section VI, this expression is equivalent to what was used in references 6 and 4.

### Case B: Uniform Stretching

Here we choose

$$\tilde{R}_p(E, x_2) = C(x_2) R_p(E) \quad (17)$$

with the constant C chosen such that

$$x_2 = C R_p(E_B) \quad (18)$$

Equation (8) than gives

$$R_p(E_1) = 1 - \left( x_1/x_2 \right) R_p(E_B) \quad (19)$$

as will be discussed in Section VI, this expression is equivalent to what was used in references 7, 8, and 9. The corresponding energy distribution was used by Marwick (8) to calculate PKA recoil spectrum.

### Case C: Fluctuations Proportional to Range Straggling

Here we choose

$$\tilde{R}_p(E, x_2) = R_p(E) + C(x_2) \alpha(E), \quad (20)$$

with the constant C chosen such that

$$x_2 = R_p(E_B) + C \alpha(E_B). \quad (21)$$

In this case the model fluctuations are proportional to range straggling

$$\tilde{R}_p(E, x_2) - R_p(E) = C \alpha(E) \quad (22)$$

Equation (8) becomes

$$x_2 = x_1 + R_p(E_1) + \left( \alpha(E_1)/\alpha(E_B) \right) \left( (x_2 - R_p(E_B)) \right). \quad (23)$$

### Energy Deposition Profiles

As a beam particle traverses the target, it deposits energy into elastic collisions given by (2).

$$S_n(E) = (dE/dR)_{\text{elastic}} = \int_0^\infty T d\sigma_n, \quad (24)$$

where T is the target atom recoil energy, and  $d\sigma_n$  is the atomic section for elastic collisions. The corresponding quantity for  $(dE/dx)$  can be approximated as

$$(dE/dx)_{\text{elastic}} = S_m(E) (dR(E)/dR_p). \quad (25)$$

The profile  $S_p(x)$  for energy deposited into elastic collisions by the entire ion beam as it traverses the depth  $x$  can be written

$$S_{B1}(x) = \int_0^{\infty} dE g(E, x) S_m(E). \quad (26)$$

A more refined approximation can be obtained by employing the expression (25) (Reference 3):

$$S_{B2}(x) = \int_0^{\infty} g(E, x) S_m(E) \left( \frac{dR(E)}{dR_p} \right). \quad (27)$$

A still more refined approximation can be obtained by including the LNST theory for energy partition (5) to obtain (3)

$$S_D(x) = \int_0^{E_B} dE g(E, x) S_L(E) \left( \frac{dR(E)}{dR_p} \right), \quad (28)$$

where

$$S_L(x) = N \int T \eta(t) d\sigma \quad (29)$$

and  $\eta(T)$  is the LNST partition function.

On using the relation (7), the above energy disposition profile becomes:

$$S_B(x_1) = \int_{x_1}^{\infty} dx_2 f(x_2) S_m(E_1), \quad (30)$$

$$S_{B2}(x_1) = \int_{x_1}^{\infty} dx_2 f(x_2) S_m(E_1) \left( \frac{dR(E)}{dR_p} \right), \quad (31)$$

and

$$S_D(x_1) = \int_{x_1}^{\infty} dx_2 f(x_2) S_L(E_1) \left( \frac{dR(E_1)}{dR_p} \right). \quad (32)$$

For case A,  $S_B$  of equation (29) becomes the energy deposition profile calculated by Kulcinski, Laidler, and Doran (6), while  $S_{B2}$  and  $S_D$  of equations (31) and (32) become the quantities calculated by Manning and Mueller with the computer code E-DEP-1 (4). For case B,  $S_D$  of equation (32) becomes the energy deposition profile calculated by Johnston, Rosalowski, Turkalo and Lauritzen (7), as described by Marwick (8) and further discussed by Matthews (9).

Marwick (8) and Matthews (9) point out that for case A one obtains a non-physical energy distribution at the target surface whereas for case B, one correctly obtains  $E = E_B$  as the only allowable energy. These results are readily confirmed by examination of equations (16) and (19) at the point  $x = 0$ . We see from equation (23) that case C also has the correct energy distribution at the surface.

Acknowledgement

The author has profited from conversations with W. A. Fraser, G. P. Mueller, and M. Rosen.

## B. Approximation for Energy Deposition in Ion Beam Bombardment

(Irwin Manning, Radiation-Matter Interaction Branch,  
Radiation Technology Division)

### Introduction

As do some other energy deposition calculations for ion beam bombardment, the NRL Code E-DEP-1 makes the approximation that a primary knockon atom (PKA) deposits its energy close to the site of creation (4). For low bombarding energies this approximation can lead to large errors, since the PKA in actuality deposits energy downstream from its creation site. Brice has formulated a method for taking those recoil effects into account (10). The present work formulates a different approach for calculating these effects by making extensive use, in the spirit of Reference (11), of a table of range distributions, the present work makes different (and probably milder) approximations than does Brice.

### Notation for Elastic Collisions

As in Reference (11), we consider a beam of ions with atomic number  $Z_1$  and energy  $E_B$  bombarding, in slab geometry, an amorphous target of atoms with atomic number  $Z_2$ . To distinguish binary collisions between beam-target atoms from binary collisions between target-target atoms, we shall denote the former by subscript 12 and the latter by 22.

We shall write the elastic cross section as  $K_{12}(E, T)$ , where  $E$  is the energy of the ( $Z_1$ ) beam atom, and  $T$  is the recoil energy of the ( $Z_2$ ) target atom. We have, for the penetrating beam

$K_{12}(E, T) dT dR =$  number of  $Z_2$  recoils of energy  $dT$  about  $T$  produced per unit time as an ion beam of unit fluence and energy  $E$  traverses a path length  $dR$  through a unit perpendicular area. (1)

For an ensemble of penetrating ions  $Z_1$ , we also have

$K_{12}(E, T) dT dR =$  ensemble average of the number of recoils of energy  $dT$  about  $T$  produced as a penetrating ion  $Z_1$  of energy  $E$  traverses a path length  $dR$  (2)

For the elastic energy loss we have

$$S_{n_{12}}(E) = (dE/dR)_{\text{elastic}} = N \int dT T K_{12}(E, T) \quad (3)$$

where  $N$  is the density of target atoms.

Let  $\phi(E, x)$  be the energy distribution of the penetrating ( $Z_1$ ) beam defined in Reference 11 and let  $\psi(T, x)$  be the distribution function for PKA's (that is, recoils produced in 12 collisions) produced by this beam:

$$\psi(T, x) dT = \text{number of PKA's of energy } dT \text{ about } T \text{ produced per unit time per unit bombarding beam flux as an ion beam with distribution } \phi(E, x) \text{ traverses a perpendicular unit area located at depth } x. \quad (4)$$

We have

$$\psi(T, x) = \int dE \phi(E, x) K_{12}(E, T) dR_{12}(E)/dR_p. \quad (5)$$

### Energy Deposition by a PKA

Consider a PKA created at the point A at depth  $x_1$  with energy  $T_1$  and velocity direction  $\hat{v}_1$ . We will assume no range straggling for  $Z_2$  atoms. Our model has range and energy straggling only for the beam ( $Z_1$ ) atoms. We also assume that the PKA travels in a straight line, coming to rest at a point C. Let B be an intermediate point at depth  $x_2$ , where the PKA has energy  $T_2$ . Let  $\mu$  be the cosine of the angle between  $\hat{v}_1$  and the x-axis:

$$\mu(E_1, T_1) = \hat{v}_1 \cdot \hat{x}; \quad (6)$$

the kinematics of elastic collision determines  $\mu$  as a function of  $E_1$  and  $T_1$ , where  $E_1$  is the energy of the  $Z_1$  beam ion creating the PKA. We have

$$x_2 - x_1 + \mu(E_1, T_1) R_{P22}(T_2) = R_{P22}(T_1) \quad (7)$$

This equation determines  $T_2$  as a function of  $T_1$  and  $E_1$ :

$$T_2 = T_2(E_1, T_1) \quad (8)$$

Now let  $S_p(x_2, x_1, T_1) dx_2$  be the damage energy deposited by this PKA as it traverses a depth  $dx_2$  around the point B. We have

$$\begin{aligned} S_p(x_2, x_1, T_1) &= S_p(x_2 - x_1, T_1) \\ &= \left( 1/\mu(E_1, T_1) \right) \eta_{22}(T_2) S_{m22}(T_2) \left( dR_{22}(T_2)/dR_p \right), \end{aligned} \quad (9)$$

where  $\eta_{22}(T)$  is the energy partition factor of Lindhard, Nielsen, Scharff, and Thompson(1).

### Energy Deposition by the Bombarding Beam

For a bombarding beam, the damage energy deposited at the point  $x_2$  is obtained by multiplying the expression (9) by the number of PKA's of energy  $T_1$  produced at  $x_1$  and summing over  $T_1$  and  $x_1$ . We will denote by  $S_{DR}(x_2)$  the damage energy including recoil effects:

$$S_{DR}(x_2) = \int_0^{x_2} dx_1 \int_{E_d}^{T_m} dT_1 S_P(x_2, x_1, T_1) \psi(T_1, x_1). \quad (10)$$

Equation (5) gives

$$S_{DR} = \int_0^{x_2} dx_1 \int_E^{T_m} dT_1 \int dE_1 S_P(x_2, x_1, T_1) \phi(E_1, x_1) K_{12}(E_1, T_1) \left( \frac{dR_{12}(E_1)}{dR_P} \right) \quad (11)$$

Let  $x_3$  be the stopping depth of the  $Z_1$  beam ion which creates the PKA of energy  $T_1$  at depth  $x_1$ ; this  $Z_1$  ion has energy  $E_1$  at the depth  $x_1$ . Using the methods of Reference (11), we can employ the range distribution  $f(x_3)$  of  $Z_1$  ions to estimate the flux  $\phi(E_1, x_1)$  of equation (11), obtaining

$$S_{DR}(x_2) = \int_0^{x_2} dx_1 \int_{E_d}^{T_m} dT_1 \int_{x_1}^{\infty} dx_3 S_P(x_2 - x_1, T_1) K_{12}(E_1, T_1) \left( \frac{dR_{12}(E_1)}{dR_P} \right) f(x_3) \quad (12)$$

Now, interchange the order of integration of  $x_3$  and  $T_1$ , and define

$$S_M(x_2 - x_1, E_1) = \int_{E_d}^{T_m} dT_1 K_{12}(E_1, T_1) S_P(x_2 - x_1, T_1) \quad (13)$$

with  $S_P$  given by equation (9).

$S_M dx_2$  is the damage energy  $dE_M$  deposited at  $dx_2$  around  $x_2$  by all the PKA's created at depth  $x_1$  by a single beam ion  $Z_1$  with energy  $E_1$ .

Define  $S_{M2}(x_2 - x_1)$  as

$$S_{M2}(x_2 - x_1) = \int_{x_1}^{\infty} dx_3 S_M(x_2 - x_1, E_1) \left( \frac{dR_{12}(E_1)}{dR_P} \right) f(x_3) \quad (14)$$

$S_{MS}$  is the damage energy function  $S_M$  for a penetrating beam with the energy straggling given by the distribution function  $\phi(E_1, x_1)$ . We now get the desired energy profile  $S_{DR}$  of equation (12) as

$$S_{DR}(x_2) = \int_0^{x_2} dx_1 S_{MS}(x_2 - x_1). \quad (15)$$

Acknowledgment

The author has profited from conversations with W. A. Fraser, M. Rosen and G. P. Mueller.

## II. HEAVY ION AND NEUTRON DAMAGE STUDIES

### A. The Effect of Alloy Additions on the Swelling of Neutron-Irradiated Nickel

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#### Background

Swelling (or volume change) of materials caused by high-fluence neutron irradiation at elevated temperatures is a major engineering design problem that limits the performance of advanced nuclear reactors. This swelling is caused by the agglomeration of radiation-produced vacancies into microscopic cavities or voids. The design of new low-swelling alloys for nuclear applications requires fundamental knowledge of the effect of individual alloying elements in simple binary alloy systems, since individual effects cannot be readily studied in the more complex commercial alloys. The present study was undertaken to examine the effects of small additions of aluminum, molybdenum, silicon, and titanium on void formation in neutron-irradiated nickel. These data were then compared with the results of previous work (12) that involved nickel-ion irradiation of the same set of alloys. This comparison provides insight into the effects of alloying on both the swelling of nickel and the correlation between neutron and heavy-ion damage.

#### Progress

The pure nickel control material and the nickel alloys used in the present study were taken from the alloy series previously examined following nickel-ion irradiation (12). The alloys Ni-1 at.%Al, Ni-1 at.%Mo, Ni-1 at.%Si, and Ni-1 at.%Ti were selected for the first examinations following neutron irradiation. The ion irradiation experiments had shown that the swelling in Ni (at 8.1 dpa\* 625°C) was reduced by a factor of 40 by the Al addition, and that significant void formation was completely suppressed by the Mo, Si, and Ti additions.

The alloys and the control material were all prepared by arc-melting ~65-gram buttons in an argon atmosphere, which had been purified by titanium gettering. After several remelts, a 3-mm thick slice was sectioned from the center of each button. The slices were cold-rolled to 0.1-mm thick foil, from which 3-mm diameter disks were punched. With the exception of the Al alloy, these specimens were annealed for one hour at 990°C in a  $1 \times 10^{-6}$  torr vacuum. The Ni-1%Al specimens were similarly annealed, but the temperature was reduced to 800°C to reduce the loss of Al from the foils.

\* The original reference calculated dpa values using a displacement energy of 25 eV for nickel, while the present report uses 40 eV to conform with current convention.

The disks were irradiated in the Experimental Breeder Reactor No. II (EBR-II) fast reactor in Idaho Falls, Idaho, to a neutron fluence of  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>,  $E > 0.1$  MeV, at a temperature of 455°C. Following irradiation, disks were thinned by dual-jet electropolishing in a solution of 250 ml methyl alcohol, 150 ml n-butyl alcohol, and 10 ml perchloric acid at a temperature of -60°C and a voltage of 35 V. Void and dislocation structures were studied by transmission electron microscopy.

The dislocation structures in all of the materials consisted primarily of tangles of line dislocations. This is illustrated in Fig. 1, which shows the structure observed in the pure Ni. As is normally observed in specimens containing voids, the dislocation lines tended to interconnect the voids.

The qualitative features of the void distributions observed in Ni, Ni-1%Al, Ni-1%Mo, Ni-1%Si, and Ni-1%Ti are shown in Figs. 2 - 6, respectively. Quantitative parameters measured from these void structures are given in Table 1. The following features of void formation in these materials can be seen:

1. The void size distribution in the pure Ni was very broad, and the voids were formed heterogeneously, with clusters of small voids dispersed among larger ones.
2. In the Ni-Al alloy, the void size distribution was narrower, and the spatial distribution of the voids was more homogeneous, but the average size, number density, and swelling were similar to the pure Ni.
3. The Mo and Ti additions both increased the swelling by approximately a factor of two, but by different mechanisms. The Mo increased the void nucleation but did not affect the void growth, leading to a higher density of similarly-sized voids. The Ti, on the other hand, did not appear to alter the void nucleation, but increased void growth, leading to a similar density of larger voids.
4. The Si addition produced the only reduction in swelling of the four alloying elements, decreasing it by approximately a factor of ten. The Si did not significantly change the void number density, but considerably reduced the mean void size.

These observed alloying effects on void formation differ significantly from the previously reported nickel-ion experiments on the same alloys (12). As noted above, all four of the alloying elements reduced or completely suppressed the swelling in the Ni-ion-irradiated Ni, by reducing the void nucleation (assuming that the ion-irradiated specimens did not contain a high density of voids smaller than the ~1.5 nm resolution limit of the microscope). A thorough understanding of the differences between neutron and nickel-ion irradiations of these alloys would require a much more extensive investigation of irradiation temperatures and damage levels for both irradiating particles. Experimental and theoretical models which are currently available, however, offer some potential explanations. A study of the swelling produced in pure Ni

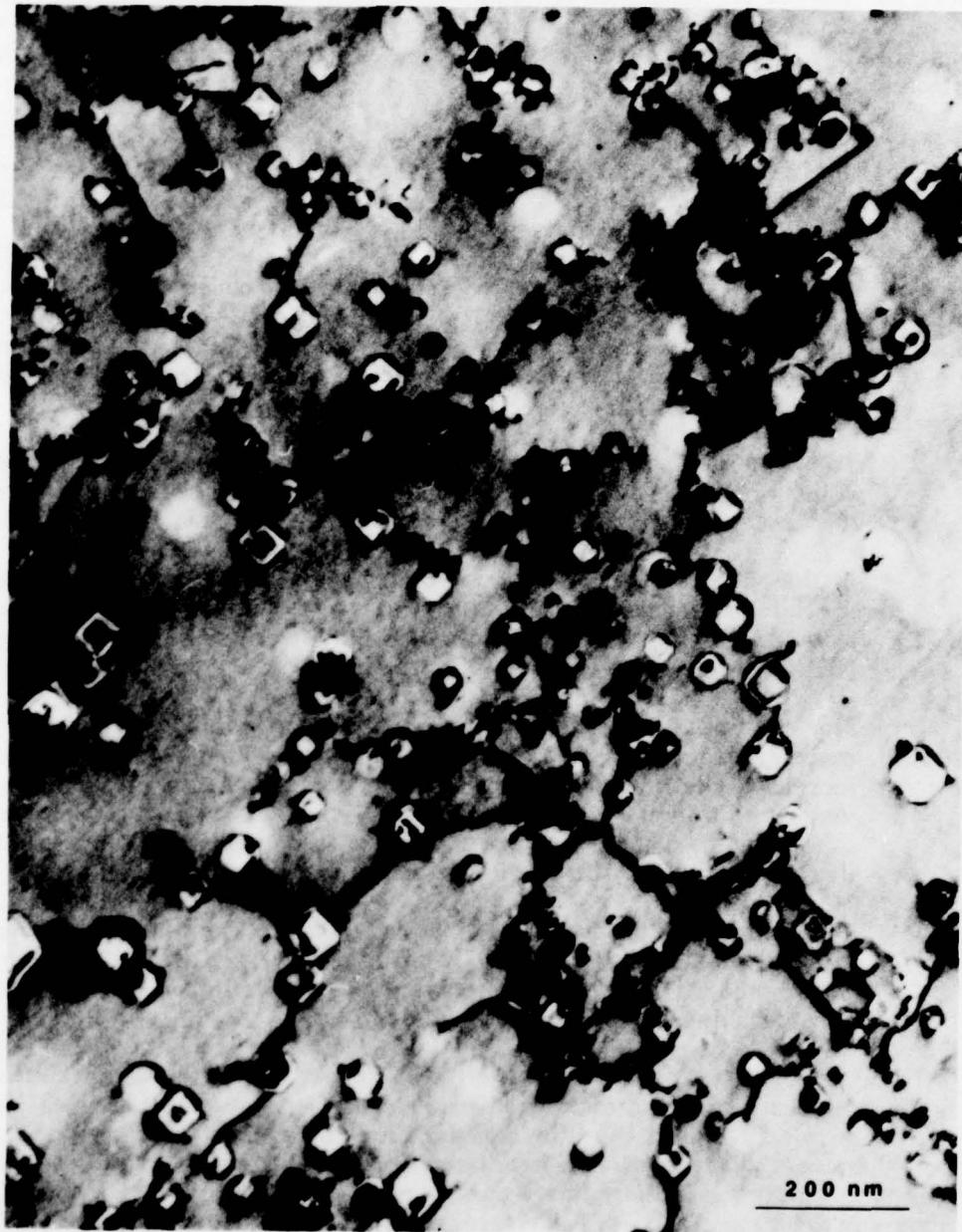


Fig. 1

The dislocation structure observed in pure nickel following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>,  $E > 0.1$  MeV, at 455°C. The dislocation structures observed in Ni-1 at.%Al, Ni-1 at.%Mo, Ni-1 at.%Si, and Ni-1 at.%Ti were qualitatively similar.

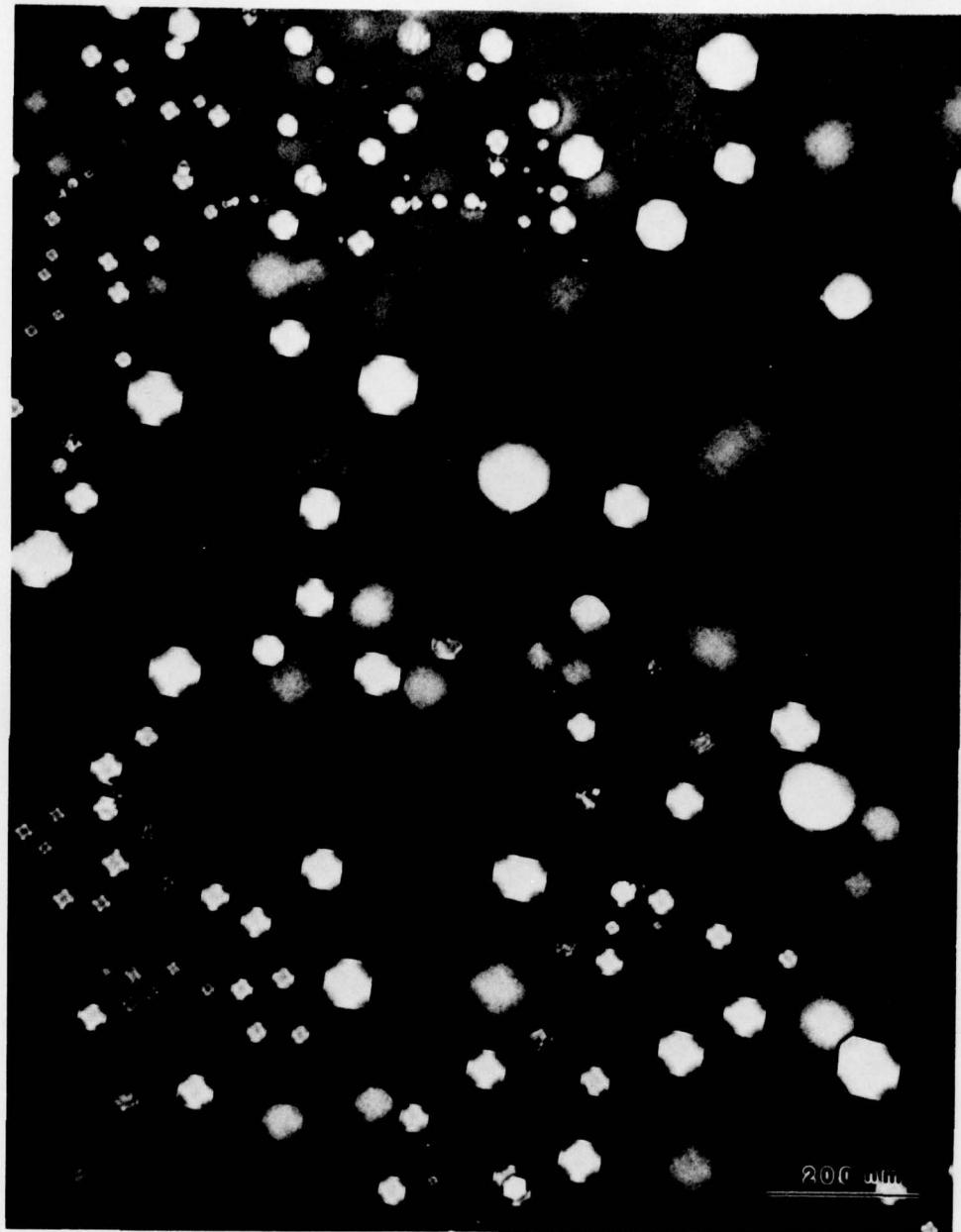


Fig. 2

Void distribution observed in pure Ni following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>,  $E > 0.1$  MeV, at 455°C.

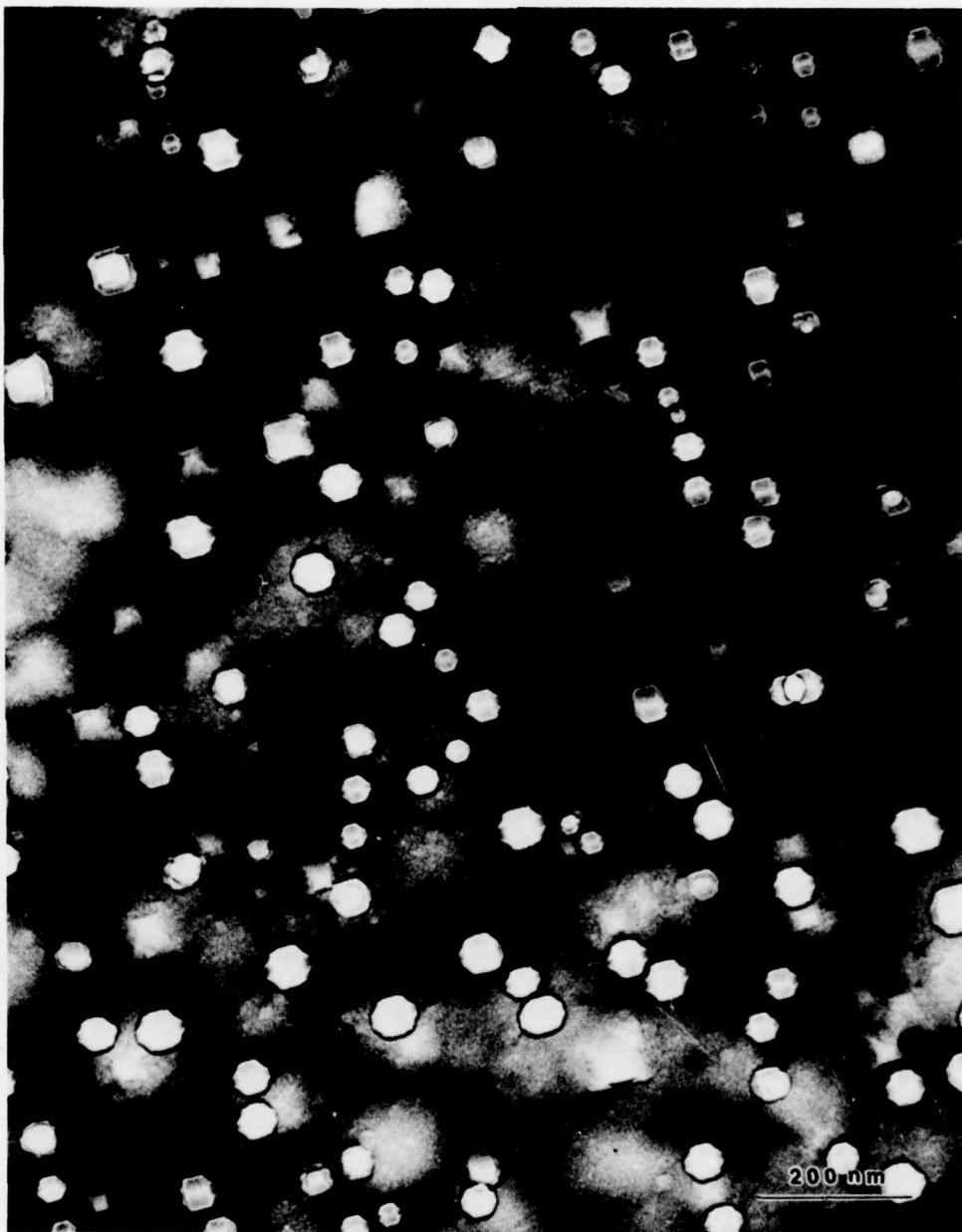


Fig. 3

Void distribution observed in Ni-1 at.%Al following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV, at 455°C.



Fig. 4

Void distribution observed in Ni-1 at.%Mo following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV, at 455°C.

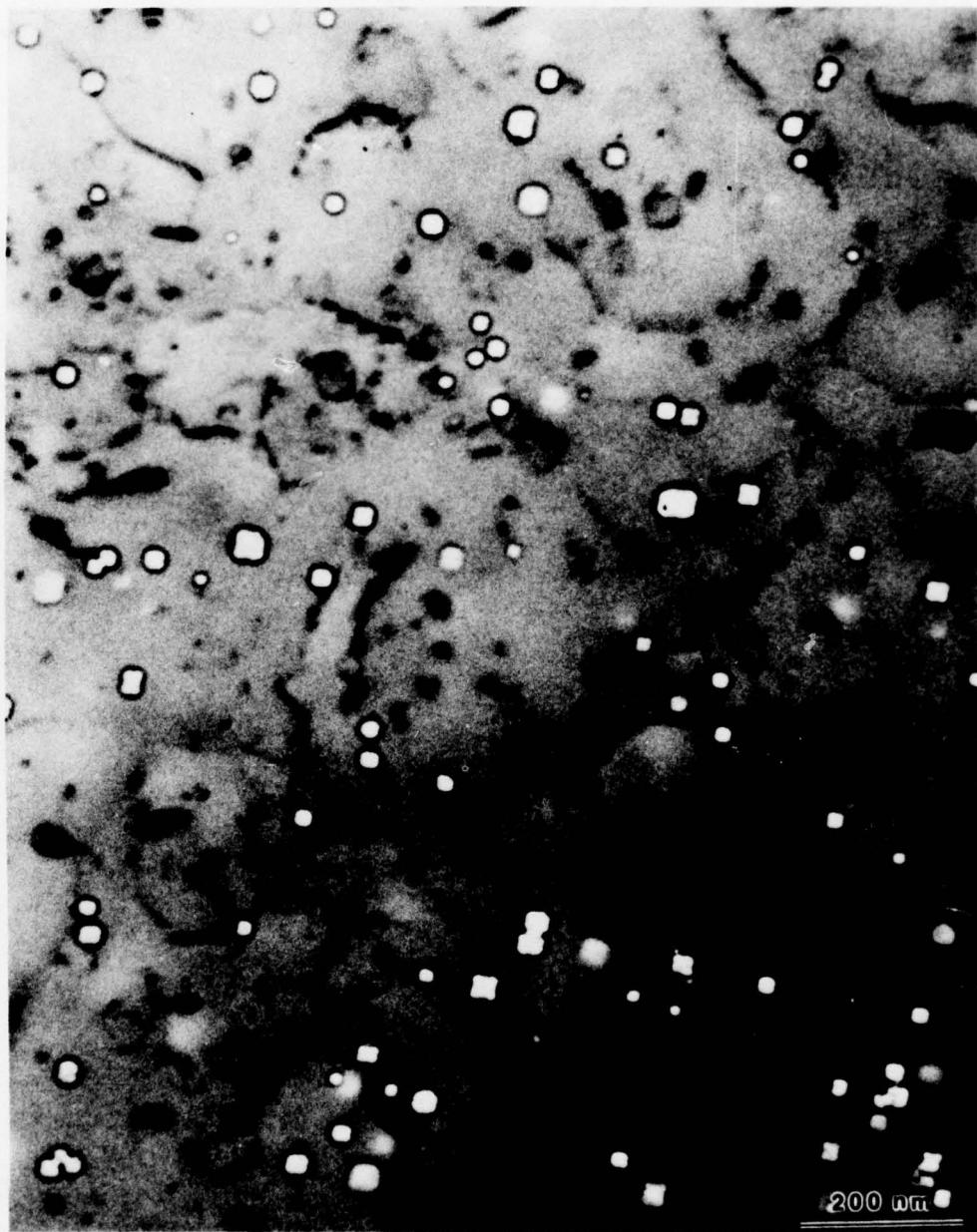


Fig. 5

Void distribution observed in Ni-1 at.%Si following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV, at 455°C.



Fig. 6

Void distribution observed in Ni-1 at.%Ti following irradiation with  $2.2 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV, at 455°C.

TABLE 1  
VOID FORMATION IN NICKEL ALLOYS  
( $2.2 \times 10^{22}$  n/cm<sup>2</sup>, 455°C)

Material	Void Density (cm <sup>-3</sup> )	Mean Void Dia (nm)	Swelling (%)
Ni Control	$3.5 \times 10^{14}$	43.5	2.56
Ni-1% Al	$4.2 \times 10^{14}$	48.8	3.04
Ni-1% Mo	$1.33 \times 10^{15}$	41.0	5.64
Ni-1% Si	$3.8 \times 10^{14}$	21.0	0.27
Ni-1% Ti	$2.8 \times 10^{14}$	62.1	4.91

by neutron and Ni-ion irradiations (13) concluded that different void nucleation mechanisms were controlling void formation in the two cases. In the ion irradiation experiments, the displacement rate was  $4.4 \times 10^{-2}$  dpa/sec,\* which produces large supersaturations of vacancies and interstitials, which can lead to significant void nucleation in the absence of insoluble gasses (14). In a fast reactor irradiation, however, displacement rate is  $\sim 1 \times 10^{-6}$  dpa/sec, and the helium produced by transmutation reactions is probably responsible for driving the void nucleation. It is therefore possible that all four alloying elements in this study could greatly reduce the homogeneous nucleation rate in a Ni-ion irradiation while either having no effect on the helium-assisted nucleation mechanism that is dominant in a neutron irradiation, or even enhancing this mechanism. A second factor which could have influenced these experiments is radiation-induced segregation of the alloying elements. In other studies (15), it has been found that alloying elements which trap vacancies or interstitials will not only affect the void nucleation and growth, but will also segregate with respect to internal point defect sinks, such as voids and dislocations. This segregation can change the rates at which these sinks absorb vacancies and interstitials, as well as reduce the amount of a solute element in solution in an alloy matrix. Both of these effects can alter the void formation in an alloy. The extent of radiation-induced solute segregation is a strong function of the damage rate, so that it will be very different in neutron and nickel-ion irradiations.

The present neutron-irradiation experiments, together with the previous ion-irradiation work on the same alloys, have demonstrated that low concentrations of solid-solution alloying elements can significantly change the void formation behavior of nickel. More importantly, these studies indicate that caution should be exercised in evaluating high-dose-rate ion irradiations of complex alloys. The effects of damage rate on all important processes, including nucleation mechanisms and solute segregation, must be considered if such ion irradiation experiments are to accurately reflect the relative swelling behavior of the alloys during reactor irradiations.

## B. The Microstructure of Neutron Irradiated Refractory Metals and Alloys

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### Background

Alloys based on the bcc refractory metals are of interest for high temperature nuclear systems, such as fusion power reactors, because of the high operating temperatures, and thus high power densities, possible with this class of materials. The engineering application of refractory alloys will require the expansion of our current data base on these materials and their behavior during high-fluence irradiation at elevated temperature. The present experiments, which are closely related to the work of the CORES program, were performed as part of the Fusion Materials Program of the Office of Fusion Energy, U. S. Department of Energy. The objective was to study the microstructural response of molybdenum, TZM, niobium, niobium-1% zirconium, vanadium, and vanadium-20% titanium to high fluence neutron-irradiation at 650°C. These results supplement previous work by a number of investigators to provide a basis for the more systematic and comprehensive experiments that will be required to adequately define the irradiation response of refractory metals and alloys.

### Progress

Experimental Procedures. The pure Mo (>99.95%), Nb (>99.99%), and V (>99.95%) were obtained from Materials Research Corporation in the form of 50- $\mu$ m thick foil. (Note that the above nominal purities are for substitutional solutes only.) The TZM (Mo-0.5 wt.% Ti-0.1 wt.% Zr), Nb-1 wt.% Zr, and V-20 wt.% Ti materials were rolled to 50- $\mu$ m thick foil from commercial-grade stock. Following the punching of 3-mm dia. disks, the metals and alloys were all annealed in a vacuum of  $1.3 \times 10^{-4}$  Pa for 1 hour at 1430°C, except for the V and V-20 Ti, which were annealed at 885°C. The foils were packed into close-fitting stainless steel tubes which were evacuated, back-filled with helium, and welded shut. The foil tubes were loaded into sodium-filled heat pipe-controlled irradiation capsules (16) for irradiation in the EBR-II reactor. Two capsules were used in the present experiments, both designed to operate at 650  $\pm$  10°C. The first (EBR-II subassembly X-200), which contained all materials except the V-20 Ti, was irradiated in Row 7 to a fluence of  $5.4 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV. The second capsule (EBR-II subassembly X-255) containing the Mo and V-20 Ti was irradiated in several positions, mostly in Row 2, to a fluence of  $8.4 \times 10^{22}$  neutrons/cm<sup>2</sup>, E > 0.1 MeV. Assuming a 40 eV displacement energy for all the materials, the lower neutron fluence corresponds to 22 dpa for Mo and TZM, 21 dpa for Nb and Nb-1 Zr, and 24 dpa for V. The higher neutron fluence corresponds to 35 dpa for Mo and 38 dpa for V-20Ti. Following irradiation, the foils were electrochemically thinned and examined by transmission electron microscopy.

## Results and Discussion

The experimental results will be discussed in each of the following subsections by comparing each pair of pure metal and alloy, in turn. The quantitative void distribution data for those materials that exhibited significant void formation are all collected in Table 2, and will be referenced in each subsection, as needed.

**Mo and TZM.** The microstructures of the Mo at both fluences and the TZM at the lower fluence were similar, consisting of imperfectly ordered void lattices and moderate densities of dislocation loops and line segments. Examples of these microstructures are shown in Fig. 7. The total dislocation density in each of the specimens examined was in the range  $5 - 7 \times 10^{19} \text{ cm}^{-2}$ . A low density of globular precipitates was observed in the TZM. In the pure Mo, a void lattice parameter of 30 nm was measured for both fluences, which is in good agreement with previous lower-fluence measurements of void lattice parameter as a function of irradiation temperature (17,18). Some ordering, on approximately the same scale as in the Mo, could be detected in the TZM, but a good void lattice parameter determination could not be made, due to the relatively large size of the voids and the imperfections in the ordering. Referring to the void distribution data in Table 2, it can be seen that the swelling measured in TZM at the lower fluence was slightly greater than that in the pure Mo, although the difference was within the error limits for these determinations. At the higher fluence, where the only data were for the pure Mo, the swelling was actually smaller than at the lower fluence, although the difference was again within error limits. Both void densities and mean diameters in the Mo were similar at both fluences. The TZM contained a lower density of significantly larger voids, a number of which appeared to have coalesced from multiple smaller voids formed earlier in the irradiation. A final observation was that the voids in the pure Mo were surrounded by the strong strain fields, which were easily visible in dark field, using a  $\langle 110 \rangle g$ -vector. No strain fields of similar magnitude were observed surrounding the voids in TZM.

The present observation of the relative swelling in Mo and TZM agrees fairly well with previously reported results of other investigators, although there are significant differences in relative void sizes and number densities. At low neutron fluences ( $\sim 10^{20} \text{ n/cm}^2$ ), Bentley, et. al (19) reported significant void formation in Mo over the entire temperature range  $330 - 850^\circ\text{C}$ , while they observed significant void formation in TZM only for "intermediate" temperatures in this range. At higher fluences of  $2.5 - 4.4 \times 10^{22} \text{ n/cm}^2, E > 0.1 \text{ MeV}$ , Bentley and Wiffen (17) observed comparable swelling in Mo and TZM at  $425$  and  $585^\circ\text{C}$ , and slightly greater swelling in TZM at  $790$  and  $1000^\circ\text{C}$ . The void number densities observed in pure Mo in the present study are consistent with the temperature dependence of void number density reported by Bentley and Wiffen. There is a significant difference, however, between the void population reported by Bentley and Wiffen in TZM and those observed in the present study. Compared with pure Mo, they observed a higher density of smaller or equal size voids in TZM at all temperatures between  $425$  and  $1000^\circ\text{C}$ , while the present study found a lower density of larger voids in TZM. Another difference between the present results and those in the literature is with those reported by Sikka and Motteff (18) for irradiation to a fluence of  $1 \times 10^{22} \text{ n/cm}^2, E > 1 \text{ MeV}$ , at temperatures between  $430$  and  $1000^\circ\text{C}$ . Although this fluence corresponds to  $21 \text{ dpa}$ , the same as the present

TABLE 2  
VOID DISTRIBUTIONS OBSERVED AFTER NEUTRON IRRADIATION AT 650°C

Material	Fluence ( $n/cm^2$ , $E > 0.1$ MeV)	Void Density ( $cm^{-3}$ )	Mean Diam. (nm)	Swelling (%)
Mo	$5.4 \times 10^{22}$	$1.0 \times 10^{17}$	7.8	3.0
Mo	$8.4 \times 10^{22}$	$9.8 \times 10^{16}$	7.2	2.3
TZM	$5.4 \times 10^{22}$	$5.2 \times 10^{16}$	10.5	4.0
Nb	$5.4 \times 10^{22}$	$2.1 \times 10^{17}$	4.6	1.5
V	$5.4 \times 10^{22}$	$5.5 \times 10^{14}$	42.1	3.6

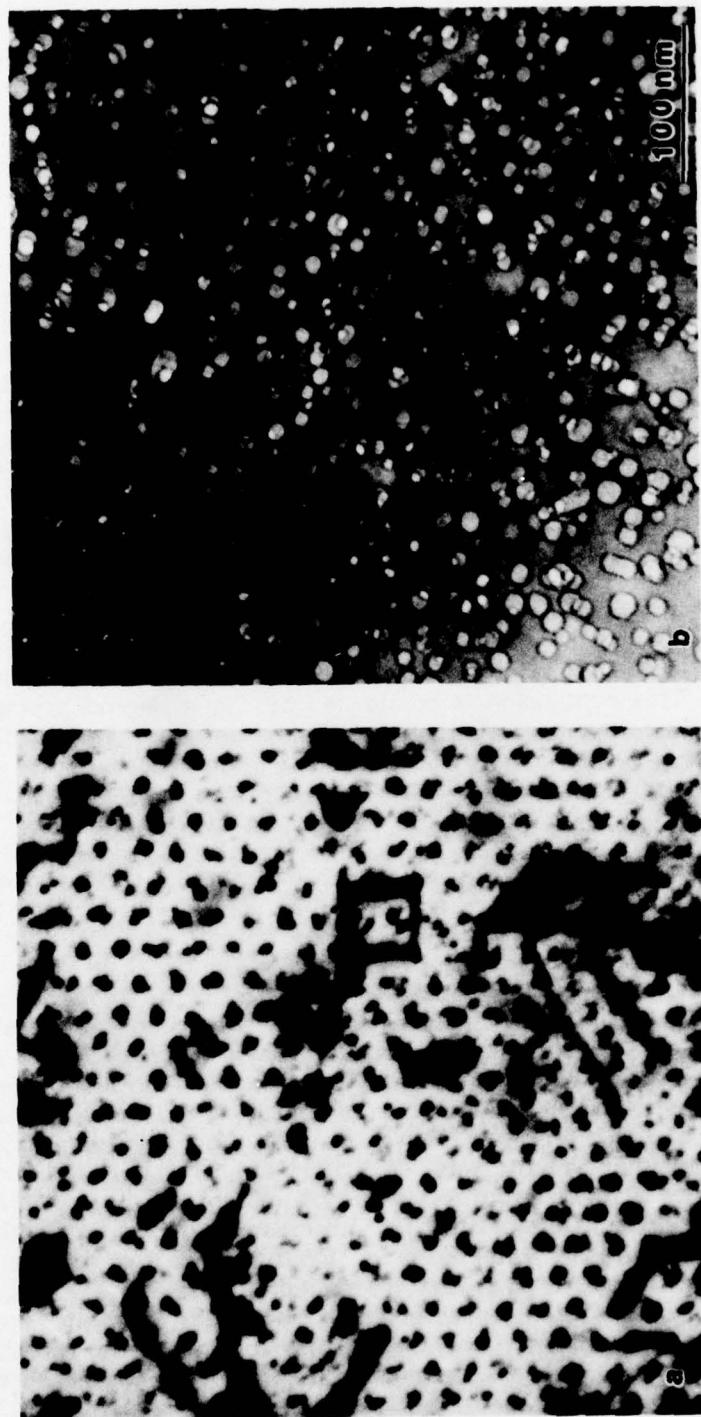


Fig. 7

Representative void structures in irradiated Mo and TZM: (a) Mo, imaged with a  $[111]$  beam direction showing void ordering; the voids appear dark due to a combination of overfocus and strain contrasts; (b) TZM, in which several coalesced voids can be seen.

study, their reported temperature dependence implies only approximately 10 percent of the presently observed swelling. The reasons for the above discrepancies are not known at this time.

Nb and Nb - 1% Zr. The Nb contained a very high density of small voids, as illustrated in Fig. 8a, as well as a much lower density of small ( $\sim 10$  nm) precipitates in the matrix. The void lattice parameter, assuming a bcc structure, was 24 nm. The swelling, as seen from the data in Table 2, was considerably less than that in the other pure metals or the TZM. Prior to irradiation, the Nb-1 Zr contained a few precipitates at the grain boundaries. After irradiation, the alloy contained additional precipitates, both at the grain boundaries and in the matrix, as seen in Fig. 8b. A few voids,  $\sim 5$  nm in diameter, were observed in isolated areas of the foil, but these did not represent any significant swelling. This observation of suppression of swelling in Nb-1Zr agrees with the results of Michel and Moteff (20) and Jang and Moteff (21), who both reported significant swelling in Nb-1Zr only over the temperature range  $700 - 900^\circ\text{C}$  at a neutron fluences of  $2.5 \times 10^{22}$  n/cm $^2$  (20) and  $5 \times 10^{22}$  n/cm $^2$  (21),  $E > 0.1$  MeV. The void distribution observed in Nb in the present study, however, was quite different from that reported by Michel and Moteff in that the voids observed here were smaller, and the number density was approximately an order of magnitude larger. The reasons for this difference cannot be determined unambiguously, but they are probably related to the impurity contents of the materials in the two studies. First, the material used in Michel and Moteff's work was commercial-grade Nb, as opposed to the high-purity stock used in the present experiment. Second, the specimens in the present study were annealed as  $50\text{-}\mu\text{m}$  thick foil in a  $1.3 \times 10^{-4}$  Pa vacuum at  $1430^\circ\text{C}$ . Although the specimen holder in the furnace was made of tantalum, considerable contamination (on the order of a few thousand parts per million) by interstitial impurities probably occurred (22). This conclusion is reinforced by the observed precipitation. The preirradiation annealing conditions for the material used in Michel and Moteff's work were not specified. It seems reasonable, therefore, that differences in substitutional and interstitial impurity contents were responsible for the different void distributions.

V and V-20% Ti. The microstructure of the irradiated pure V was quite different from those of the Mo and Nb, as shown in Fig. 9a. The preirradiation annealing treatment had produced a fine grain size ( $5 - 10\text{-}\mu\text{m}$ ), and after irradiation, each grain contained a moderate density of fairly large voids. The voids were denuded near each grain boundary. The dislocation density in the grain interiors was low,  $\sim 1 \times 10^{12}$  cm $^{-2}$ , although many dislocations were observed in subgrain boundaries. No precipitates were observed. The preirradiation microstructure of the V-20Ti contained a heterogeneous distribution of precipitates, the diffraction patterns of which were consistent with hexagonal  $\text{TiO}_2$  (rutile). The maximum observed local number density of these precipitates was  $2 \times 10^{13}$  cm $^{-3}$ , while the average density over a larger specimen volume was  $\sim 4 \times 10^{12}$  cm $^{-3}$ . A detailed characterization of the precipitate size distributions was not made, but two morphologies were observed: blocky precipitates with a 150 nm mean diameter, and rod-shaped,  $\sim 100$  nm in cross section and  $200 - 400$  nm long. Irradiation of this material at  $650^\circ\text{C}$  to  $8.4 \times 10^{22}$  neutrons/cm $^2$ ,  $E > 0.1$  MeV, produced no observable voids, but did result in significant growth of the precipitates. The mean diameter of the blocky particles increased to 500 nm; the mean cross section of the rod-shaped precipitates was 150 nm, while the maximum length of the rods increased to 1500 nm. A

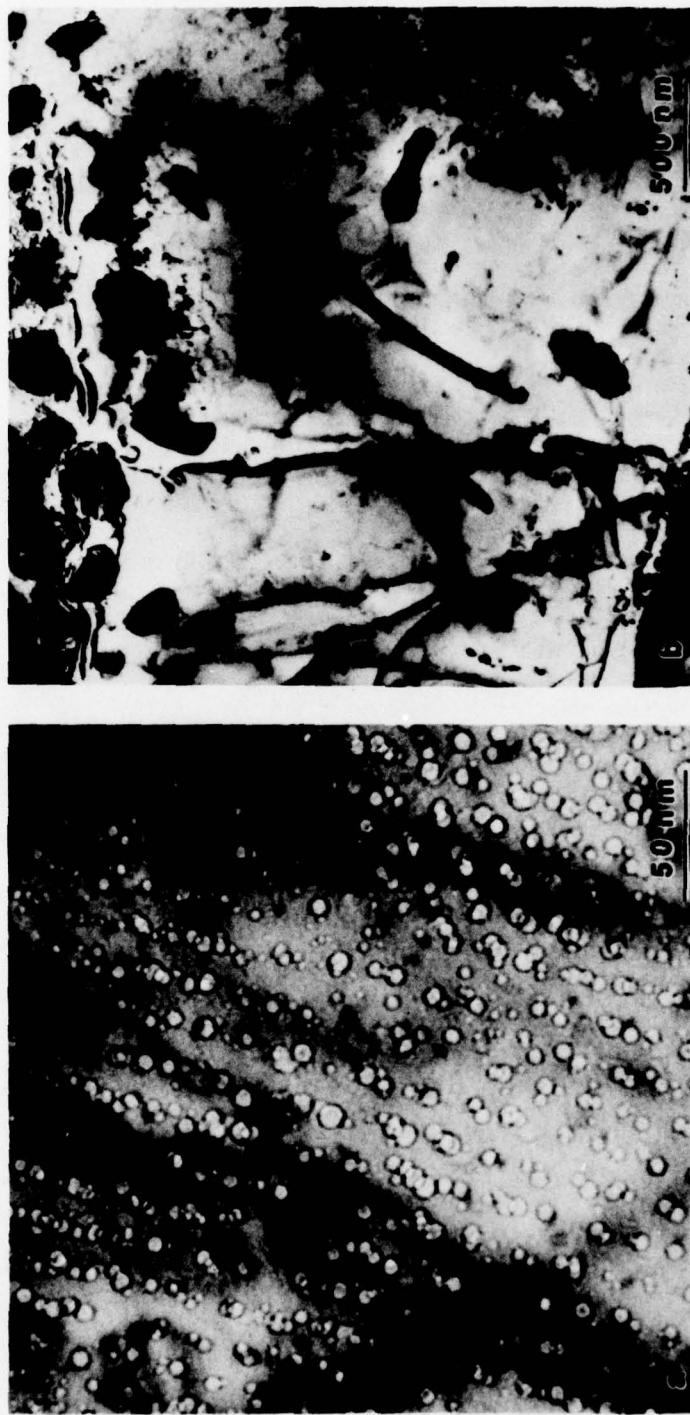


Fig. 8  
Representative microstructures in irradiated Nb and Nb-1%Zr: (a) Nb, containing a high density of small voids; (b) Nb-1% Zr, with no void formation, but extensive matrix and grain boundary precipitation. Note the different magnifications in the two micrographs.

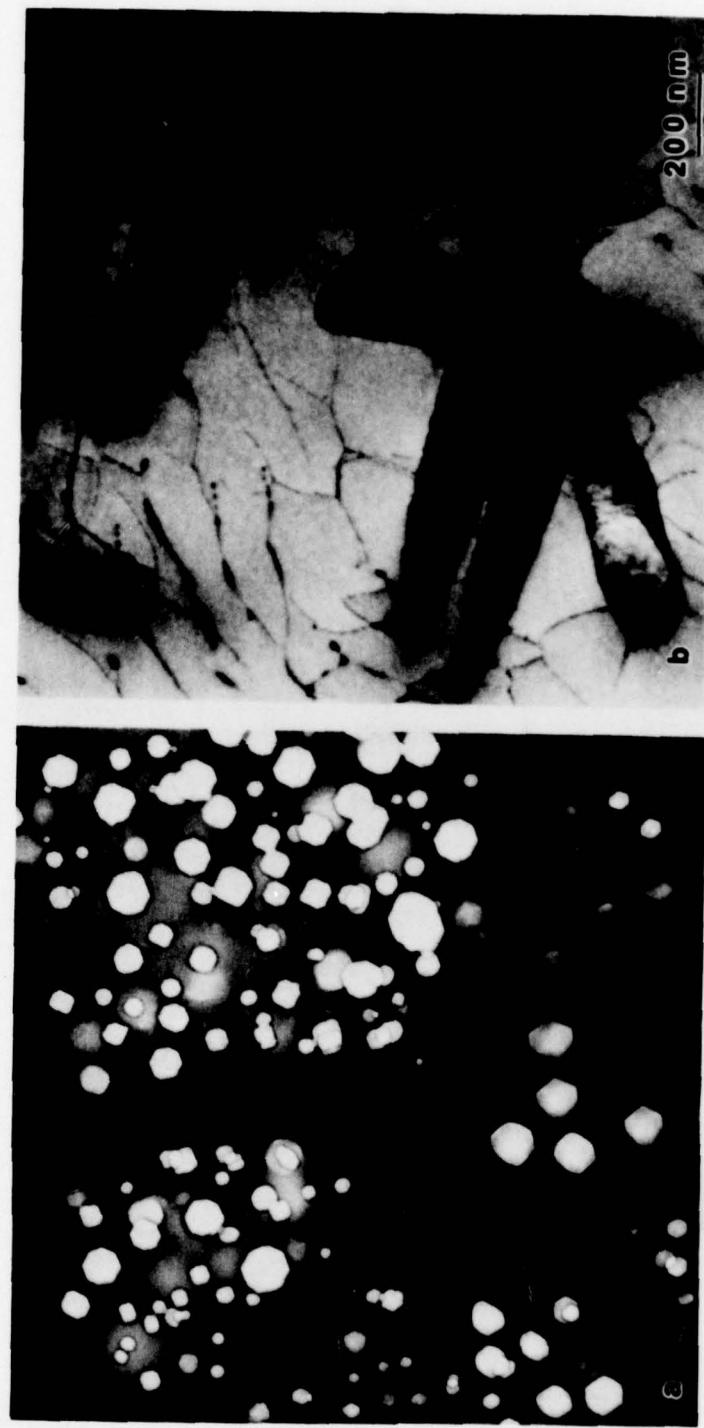


Fig. 9

Representative microstructures in irradiated V and V-20% Ti: (a) V, showing the denuding of voids at boundaries of the small grains; (b) V - 20% Ti, containing elongated  $TiO_2$  precipitates which increased in maximum length from 400 to 1400 nm during irradiation.

cluster of the elongated precipitates in the V-20Ti is shown in Fig. 9b.

The complete suppression of void formation by the addition of 20% Ti to V agrees with the results of Bentley and Wiffen (23), who examined V-20Ti irradiated to fluences of  $1.3 - 6.1 \times 10^{22} \text{ n/cm}^2$ ,  $E > 0.1 \text{ MeV}$ , at temperatures between  $470$  and  $780^\circ\text{C}$ . Before irradiation, their specimens contained precipitates similar in size, shape, and maximum number density to those observed prior to irradiation in the present study, although they reported that the specimen regions containing this maximum number density represented only  $\sim 1\%$  of the specimen volume. They observed no change in this preirradiation precipitate structure after irradiation. Carlander (24) reported a few voids in V-20Ti after irradiation to  $6 \times 10^{22} \text{ n/cm}^2$ ,  $E > 0.1 \text{ MeV}$ , at  $600^\circ\text{C}$ , although these voids did not represent any significant swelling. He did not report on the precipitation behavior of this alloy, but a number of apparent rod-shaped precipitates can be seen in his published micrograph (Fig. 3 of Carlander, Reference 24).

The void microstructures in the pure V are difficult to correlate with those reported previously for lower fluence irradiations (24,25), due to differences in starting materials and irradiation temperatures, but the observation of 3.6% swelling at  $5.4 \times 10^{22} \text{ n/cm}^2$  is consistent with approximately linear swelling with fluence, when compared with the 1.54% swelling reported by Carlander (24) for irradiation to  $2.5 \times 10^{22} \text{ n/cm}^2$  at  $625^\circ\text{C}$  and the 1.47% swelling reported by Stiegler (25) for irradiation to  $1.4 \times 10^{22} \text{ n/cm}^2$  at  $600^\circ\text{C}$  (all fluences for  $E > 0.1 \text{ MeV}$ ).

### Conclusion

The present microstructural observations of refractory metals and alloys after high-fluence neutron irradiation at  $650^\circ\text{C}$  indicate that, at least at this temperature, void swelling will probably not be the most serious obstacle to the application of this class of alloy. The swelling of even the pure metals is only moderate, and with the possible exception of Mo alloys, it appears possible to control void formation further by alloying. Changes in mechanical properties due to precipitation of interstitial impurities and the formation of high densities of small voids may be far more serious problems. Theoretically, the precipitation could be controlled by reducing the concentration of interstitial contaminants, but more comprehensive studies on thoroughly characterized materials will be required to examine this possibility. If detrimental changes in microstructure can be avoided by careful control of impurities, fabrication technologies and reactor operating conditions will still have to be developed to maintain this impurity control in large structures. If these developments can be made, however, refractory metal alloys appear to be potentially valuable for high temperature nuclear applications.

### III. LIGHT ION BEAM STUDIES

#### A. Deuterium Retention in Aluminum and Stainless Steel With and Without Helium Loading

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Fast neutrons in high flux reactors generate hydrogen and helium in reactor structural materials by  $(n,p)$  and  $(n,\alpha)$  reactions. Helium is known to accumulate in metals and alloys under reactor operating conditions and its accumulation is intimately related to swelling and other changes in mechanical properties. It has generally been assumed that hydrogen, although generated in larger amounts than helium, diffuses or permeates through the material and consequently not causing any mechanical property changes. However, retention of appreciable amounts of hydrogen could lead to deleterious effects.

Lanford (26) reported that hydrogen implanted into single crystal silicon wafers was retained indefinitely but that hydrogen retention in tungsten was enhanced with helium predamage with little retention in samples of tungsten without predamage. Nowicki (27), based upon hydrogen profile measurements by Pieper at NRL, showed that large amounts of hydrogen were present in rf-spattered  $Al_2O_3$  films deposited by planer magnetron, it remained stable and affects mechanical properties. Picraux (28) has shown enhanced hydrogen and deuterium retention in molybdenum with helium predamage, both for single crystal samples and rolled foil. Since helium is known to become substitutional by falling into vacancies created by atomic displacements and positrons have been observed to attach to these helium filled vacancies, the possibility exists that hydrogen, which would normally remain interstitial and diffuse through the metal, may attach itself to these filled vacancies.

In controlled thermonuclear systems high fluxes of 14-MeV neutrons will bombard structural materials including first walls, blankets and cryogenic cooling systems. The generation of hydrogen and helium will be enhanced by the higher cross-sections and in addition large fluxes of deuterium and tritium escaping from the plasma will strike the first walls. The question of hydrogen retention being enhanced by helium loading or radiation damage is of importance, particularly at higher temperatures. Of course, the payoff will be if retained hydrogen causes changes in mechanical properties.

As a first step, samples of aluminum, stainless steel, molybdenum and tantalum were implanted with deuterium both with and without prior helium implantations. For the helium loading, a 60 MeV  $\alpha$ -particle beam from the cyclotron was degraded sufficiently to stop within the sample and due to straggling give a broad profile. A helium implant in a 1.2 mil aluminum foil was degraded to give a profile peaking at a depth of 0.5 mils as shown by the solid curve in Fig. 10. The degradation of the alpha beam was selected to give a profile peaking at 0.17 mils in a 0.6 mil stainless steel sample, see Fig. 11. A proton beam was not used as the hydrogen isotope implant because previous measurements (29) showed surface contamination of the order of micrograms of

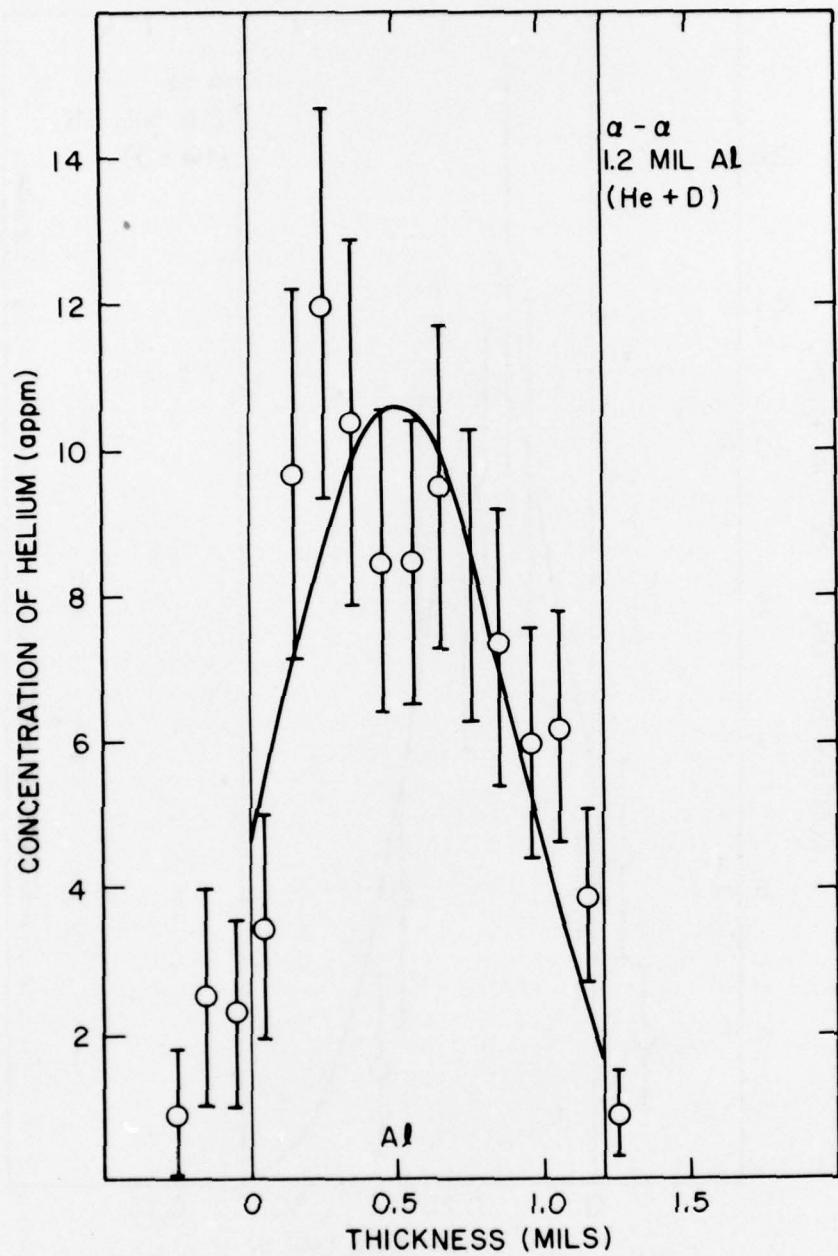


Fig. 10

Helium profile in a 1.2 mil aluminum sample implanted with helium and deuterium. The solid curve is the predicted implantation obtained from the computer code normalized by integrated current. The data points are from  $\alpha - \alpha$  scattering when a 36.5 MeV alpha beam impinges on the sample.

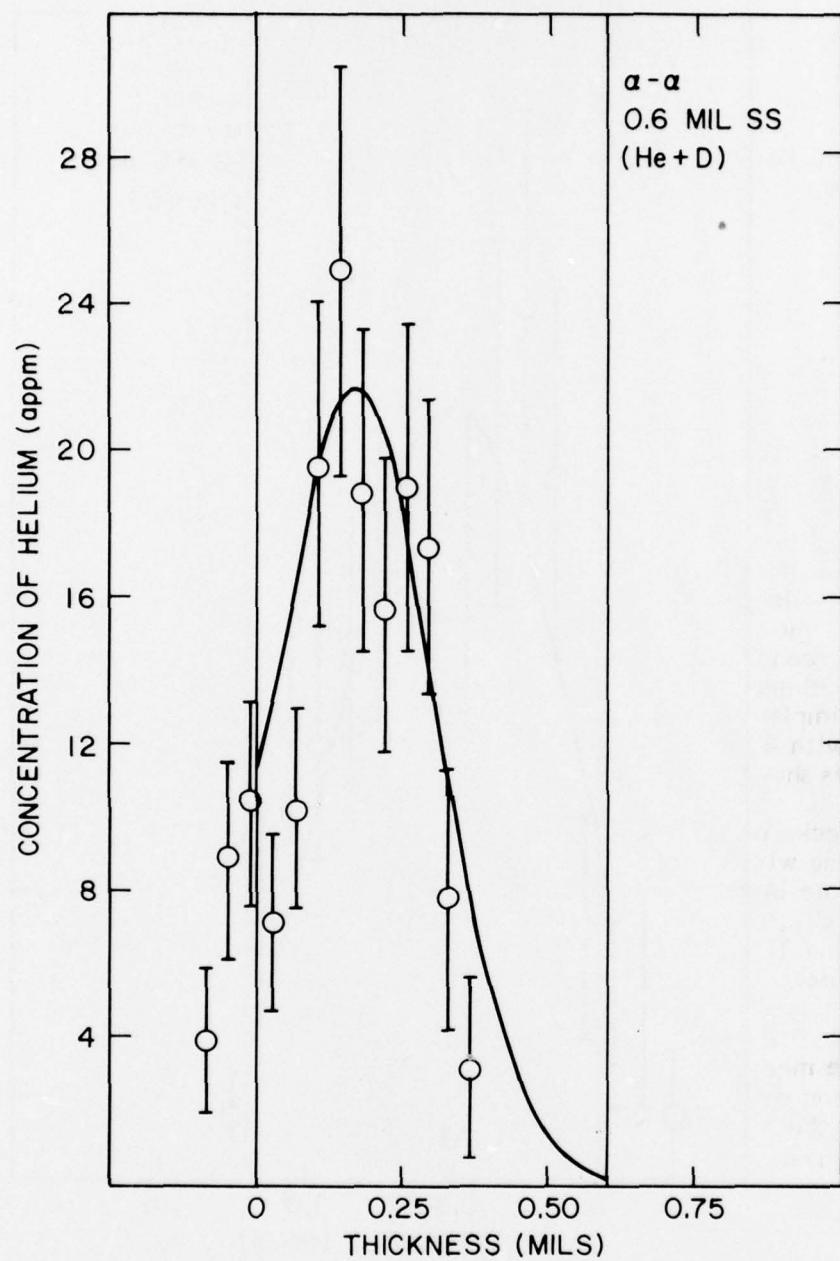


Fig. 11

Helium profile in a 0.6 mil stainless steel sample implanted with both helium and deuterium as in Fig. 1. The solid curve is the predicted helium profile.

hydrogen per  $\text{cm}^2$  complicate hydrogen profiling by p-p elastic scattering. A 22.5 MeV deuteron beam was similarly degraded to give deuteron profiles peaking at the same depths in aluminum and stainless steel. The molybdenum (2.0 mils) and tantalum (1.0 mil) were similarly loaded but these specimens were too thick to profile at the loadings. The predicted profiles are obtained from a computer code written by Pieper that gives implantation concentrations in atomic parts per million (appm) per microcoulomb of implantation beam.

The loadings of two aluminum and two stainless steel specimens are shown in Table 3. Helium and deuterium profiles were measured by scattering with a 36.5 MeV alpha beam and by d-d scattering of a 22.5 MeV deuteron beam, respectively. A measured helium profile of the Al sample implanted with both  $\alpha$ 's and d's is shown in Fig. 10 in reasonable agreement with the predicted profile normalized by the integrated current during helium implantation. Due to the low implantation levels, statistics are poor and accidental backgrounds from host scatterings generated problems. The deuteron profile for this same aluminum sample is shown in Fig. 12 along with the predicted profile. The deuteron profile for the aluminum sample not preloaded with helium is similar and agrees well with the predicted loading indicating that the deuterium is being retained in aluminum under both conditions. In the stainless steel specimens, the measured helium profile as shown in Fig. 11 agrees reasonably well with the predicted peak. However, the deuteron profiles of the two stainless steel samples are different. The peak concentration of the deuteron profile measured in case of a 0.6 mil thick specimen previously loaded with helium agrees well with the predicted damage implant as seen in Fig. 13. The profile of deuterium in the sample not loaded with helium was not very statistically significant above background effects as shown in Fig. 14.

Checks of solid angles, alignment of the apparatus and depth resolutions were made with standard helium implanted samples, both surface implants and mid-sample implants. Similar checks were made for the deuterium profiles by use of a  $\text{CD}_2$  target 5  $\mu\text{m}$  thick and with similar  $\text{CD}_2$  specimens placed in front and behind the aluminum and stainless steel samples. The surface specimens provide data to correct for multiple scattering effects occurring after the primary elastic event.

The measured peak values of the respective profiles are shown in Table 3 in comparison with the predicted implanted values. While a significant difference between the behavior of aluminum and stainless steel is evident, the experiment will be repeated with implants at least an order of magnitude higher dosages.

TABLE 3

PEAK CONCENTRATIONS OF IMPLANTED AND MEASURED PROFILES

Material	Thickness (Mils)	Implanted (appm)		Measured (appm)	
		He	d	He	d
Al	1.2	12	6	10 $\pm$ 3	5 $\pm$ 1
Al	1.2	0	7	-	6.5 $\pm$ 2.5
ss	0.6	22	10	20 $\pm$ 5	10 $\pm$ 3
ss	0.6	0	12	-	<4

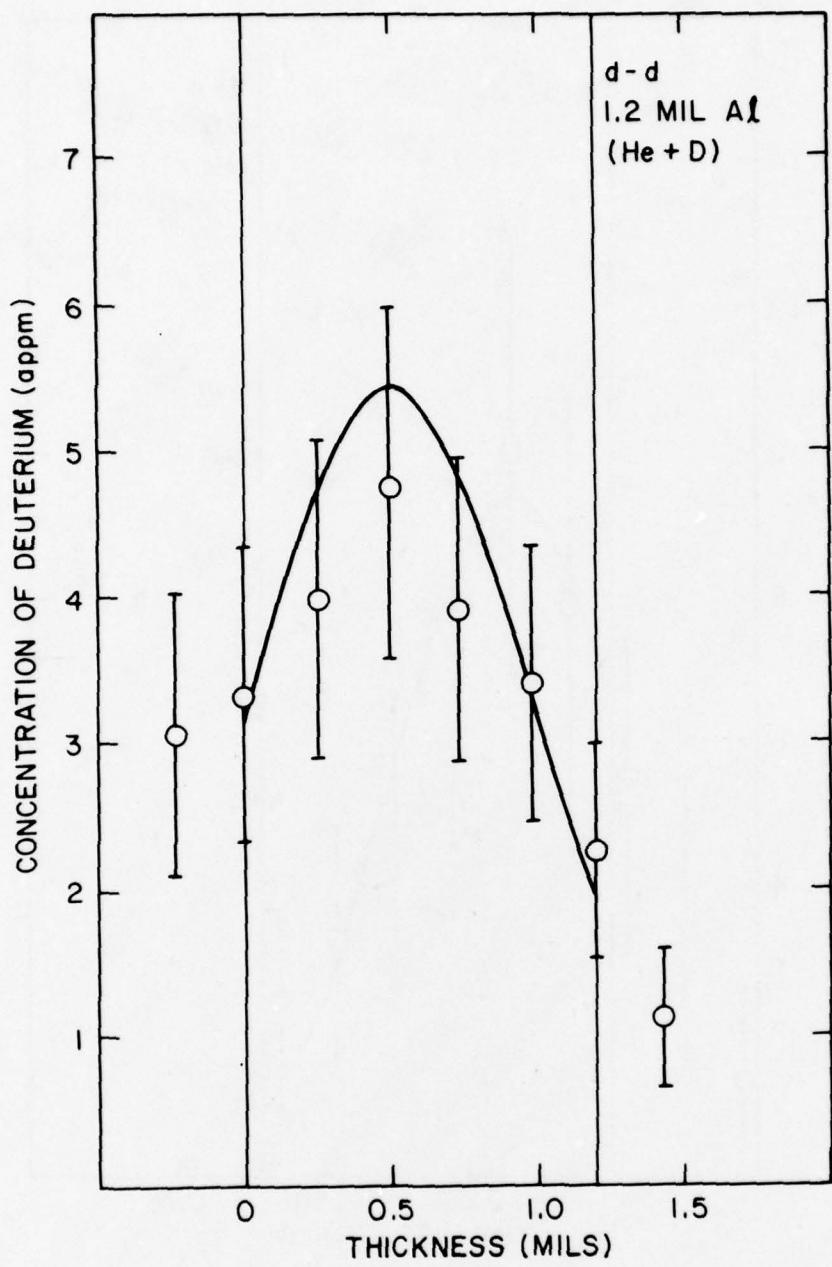


Fig. 12

Deuterium profile for the 1.2 mil aluminum sample implanted with helium and deuterium as measured by d-d scattering of a 22.5 MeV deutron base. The solid curve is the predicted deuterium profile.

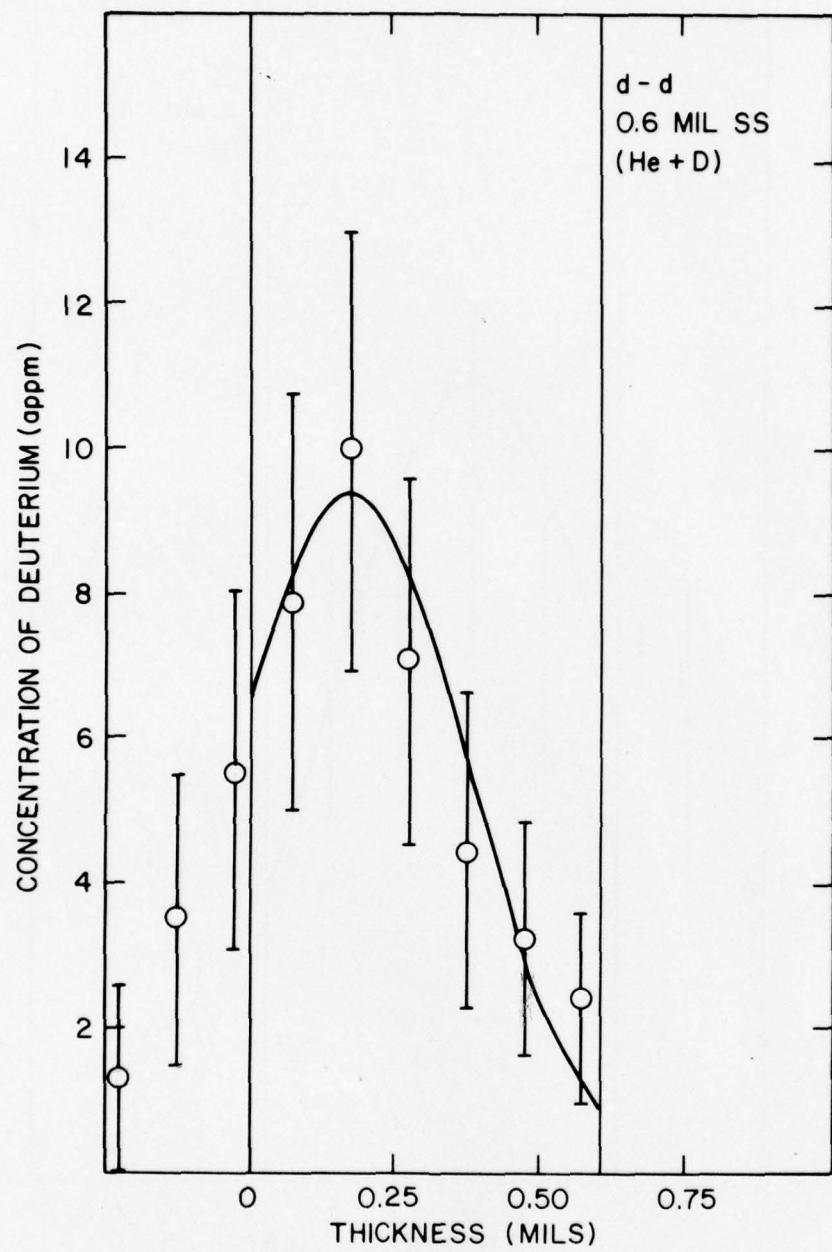


Fig. 13

Deuterium profile for the 0.6 mil stainless steel sample implanted with both helium and deuterium with the predicted curve.

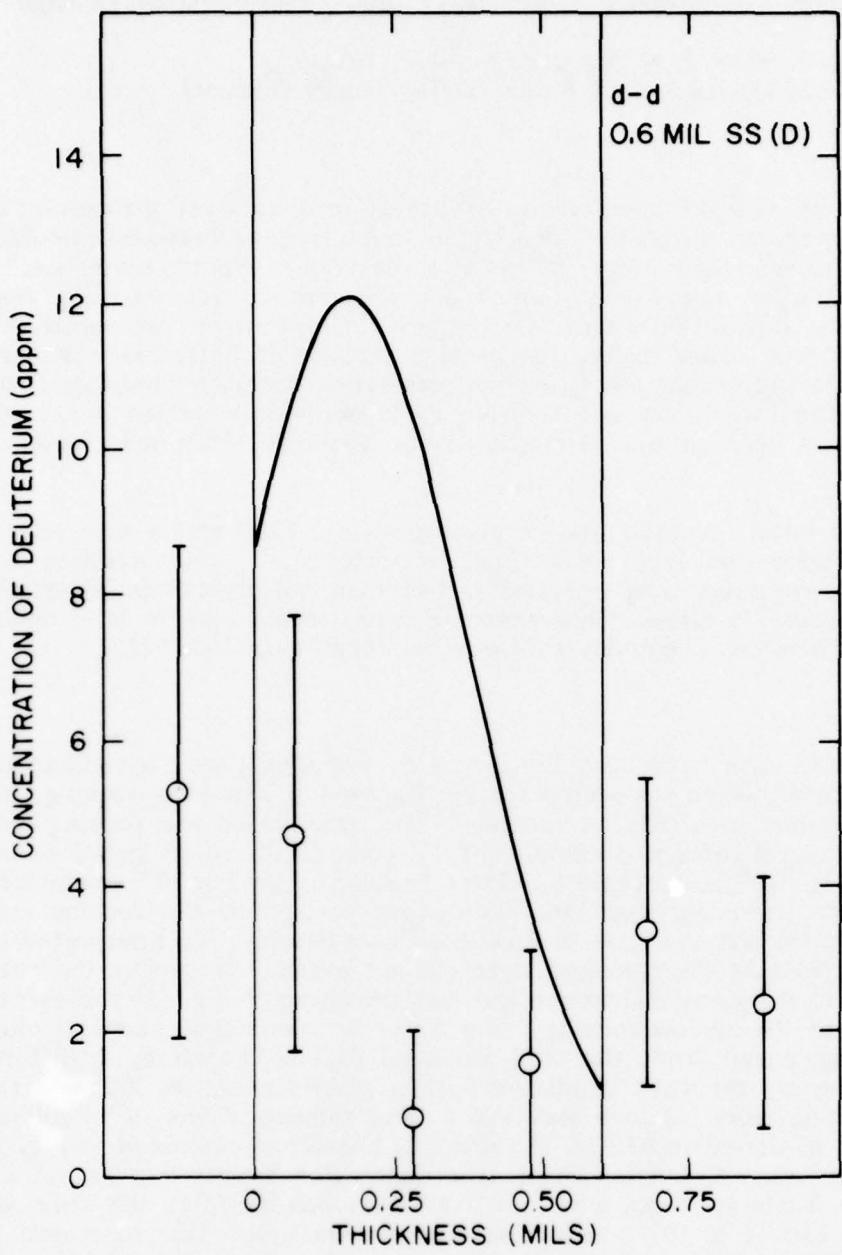


Fig. 14

Results of a deuterium profile of the 0.6 mil stainless steel sample implanted with only deuterium compared with the predicted curve.

## B. X-ray Diffraction Measurements of Low-Level Radiation Damage

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### Background

1. It is well documented in the literature that x-ray diffraction can be used to measure dislocation density in crystals and therefore should be useful in measuring early stages of radiation damage in reactor materials. We proposed that alloy compositions which are resistant to void swelling might show different diffraction characteristics than alloys which are sensitive to void swelling (viz, lower dislocation density because of better recombination of interstitials and vacancies). If such a correlation could be established, then x-ray diffraction would be an effective rapid screening method for testing proposed alloys because the irradiation time would be reduced by orders of magnitude.

For the initial experiments we planned to use Cu crystals with selected low-concentration additives whose specific effects on void swelling have already been reported. Single-crystal rather than polycrystalline material is preferred because it allows more accurate x-ray measurements in a double-crystal spectrometer. The crystals were to be prepared by Code 5220.

### Progress

Results to date have been limited to a crystal of pure Cu because it is the only specimen which has been prepared. Figure 15a shows the rocking curve obtained for that specimen as received. The area under the rocking curve (called the integral reflection coefficient, R) corresponds to an initial dislocation density of  $<10^6/\text{cm}^2$ , Table 4. After irradiation by  $2 \times 10^{-2}$  coulombs of 3.6 MeV alpha particles, Fig. 15b, the central portion of the rocking curve seems unaffected but the base is broadened considerably. We interpreted this as an indication that the damaged layer did not extend throughout the region contributing to the x-ray diffraction and that the curve of Fig. 15b represents a combination of the narrow rocking curve from the undamaged material plus a broad rocking curve from the thin damaged layer. Therefore, a different portion of the crystal was irradiated with a graded series of alpha-particle energies ranging from 1.2 to 4 MeV and a total fluence of  $2 \times 10^{-2}$  coulombs. This resulted in the curve of Fig. 15c which is broadened compared to Fig. 15a but retains a nearly Gaussian shape. The dislocation density has increased to  $3 \times 10^7/\text{cm}^2$ , Table 4. Next, a second irradiation was added to the same area as for Fig. 15c ( $2 \times 10^{-2}$  coulombs, graded energy). This increased the dislocation density, Table 4, and resulted in the rocking curve of Fig. 15d.

When dislocation density is plotted vs irradiation in Fig. 16 it appears that the damage increases at a faster than linear rate with irradiation initially but that it is beginning to approach a linear rate after  $0.04 \text{ C}/\text{cm}^2$ .

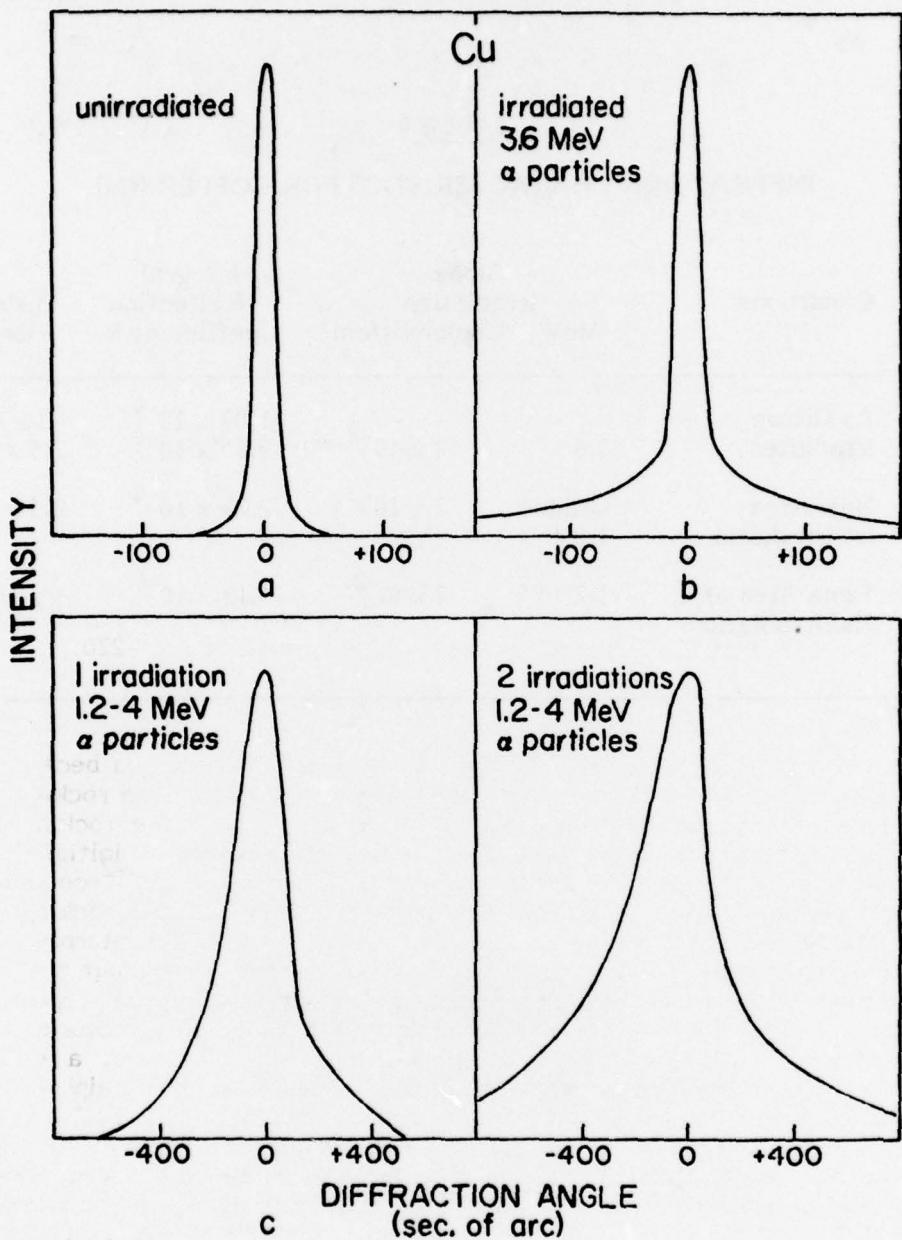


Fig. 15

Rocking curves for Cu single crystal. Irradiation with alpha particles of a single energy (b) broadens the base of the curve but alters the central peak only slightly. Irradiation with alpha particles of graded energies broadens the whole rocking curve (c and d).

TABLE 4  
 DIFFRACTION CHARACTERISTICS FOR COPPER (Cu)

Run	Conditions	Alpha Irradiation MeV	Alpha Irradiation Coulombs/cm <sup>2</sup>	Integral Reflection Coefficient,R	Dislocation Density
a	As Grown	-	-	$1.03 \times 10^{-4}$	$< 10^6/\text{cm}^2$
b	Irradiated	3.6	$2 \times 10^{-2}$	$2.65 \times 10^{-4}$	$2.5 \times 10^7$
c	New Area 1st Irradiation	Graded 1.2 to 4	$2 \times 10^{-2}$	$2.74 \times 10^{-4}$	$2.5 \times 10^7$
d	Same Area as c, 2nd Irradiation	1.2 to 4	$2 \times 10^{-2}$	$5.17 \times 10^{-4}$	$3 \times 10^8$

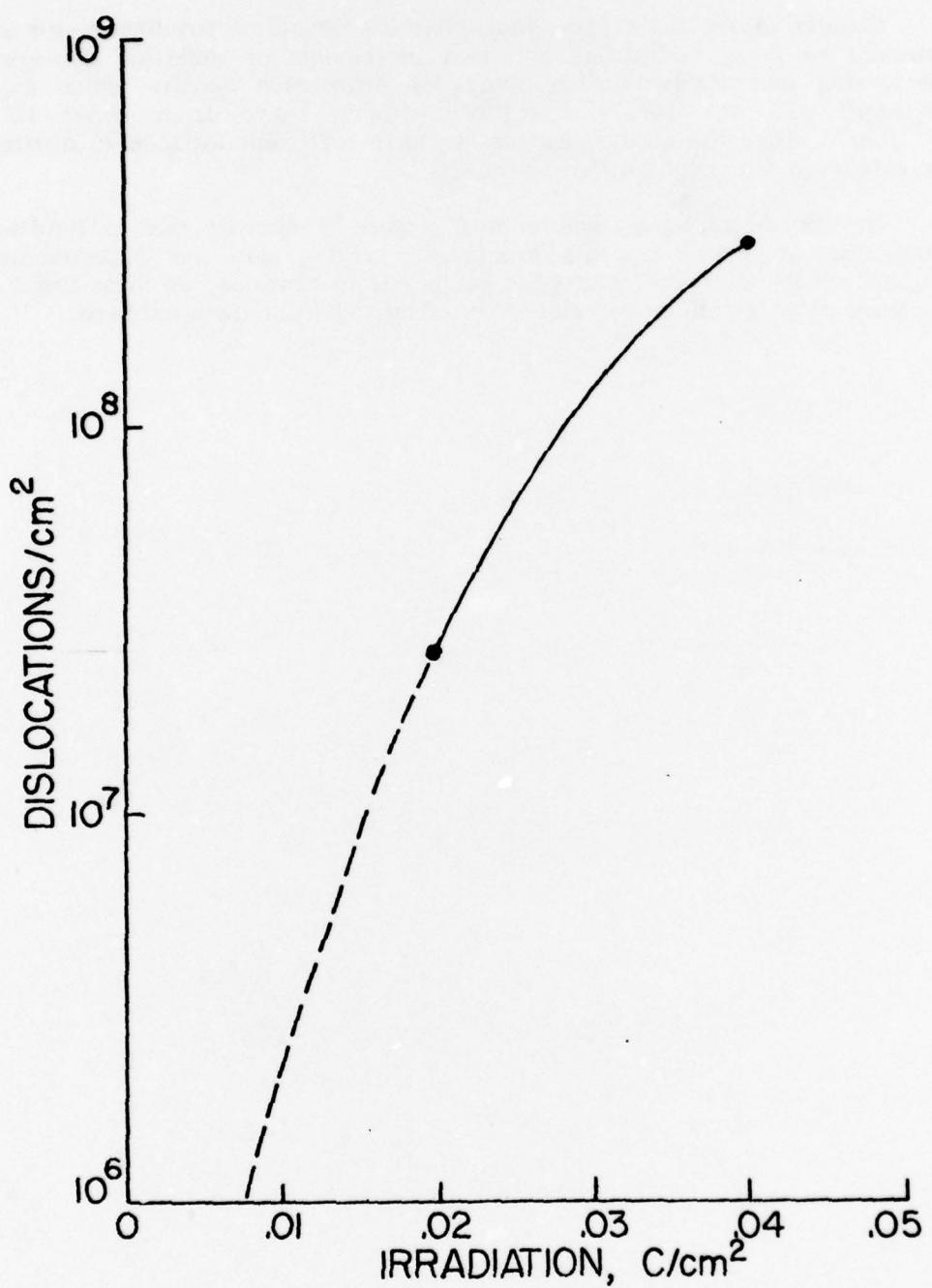


Fig. 16

Dislocation density as determined from x-ray rocking curves versus irradiation by alpha particles.

### Conclusions

Results of the Cu crystal show that low levels of irradiation are easily observed by x-ray diffraction and that increments of radiation damage are measurable quantitatively. The limits of dislocation density which can be measured with the x-ray apparatus available range from about  $10^6$  to  $10^{10}/\text{cm}^2$ . Thus the method appears to have sufficient latitude to distinguish the effects of alloying additions to the Cu.

On the discouraging side of the picture it appears that difficulties in production of single crystals precludes carrying out the experiments as originally planned. Therefore, if the project is to continue, we must find a way to utilize polycrystalline material or go outside NRL for the specimens.

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